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CAMERON STATION ALEXANDRIA. VIRGINIA

AN AUTOMATIC X-RAY DIFFRACTOMETER FOR POLE-FIGURE ANALYSIS

OF POLYMER ORIENTATION

January 15, 1965

C. Richard Desper

Submitted by
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ABSTRACT

An automated x-ray diffractometer has been designed and built
to obtain pole figure data on polymeric samples. The pole figure goni-
ometer is automatically driven, in a prefixed, constant-increment program,
by two pulse motors, allowing the operator to determine the complete distri-
bution pattern for a given h k l plane automatically. The results are
punched out on IBM cards; two programs have been written to process this
data on the IBM 1620 computer. The first program computes the x-ray in-
tensity at each sample orientation, corrected for absorption, polarization,
background, and incoherent scattering. The second program uses this in-
tensity data to obtain locations of constant-intensity contour lines.

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We wish to acknowledge the assistance of the following people, whose help made this report possible:

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AN AUTOMATIC X-RAY DIFFRACTOMETER

FOR POLE FIGURE ANALYSIS

OF POLYMER ORIENTATION*

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INTRODUCTION

During recent years the Polymer Research Institute has made greater
and greater use of x-ray diffraction as a technique for studying polymer
structure. Quite naturally, this led to the construction of an automated
x-ray diffractometer and to the use of a computer to process the raw data.[2,3,4,5]
Experiments with this apparatus yielded valuable data on polymer crystallization
and orientation.

When it was decided to extend this work to include measurement of
biaxial orientation by x-ray diffraction, a second automated diffractometer
was designed and built specifically for these experiments. The purpose of this
report is to describe the resulting apparatus, including both the diffractometer
and its associated control unit.

II. X-Ray Diffractometer

A. <u>General Layout</u>

The general layout of the biaxial scan diffractometer is shown in Figure 1.
The entire apparatus is bolted to a $\frac{1}{2}$ inch thick aluminum plate, which rests on
top of the x-ray high voltage generator. The x-ray tube is mounted in a standard
North American Phillips tube tower; however, the tube tower is in a horizontal
position instead of its normal vertical position. To support the tube tower, a
tube stand, shown in Figure 2, was fabricated. The tube tower bolts to the up-
right member of the stand, and the high voltage cable connection is supported by
two $\frac{1}{4}$ inch thick aluminum "wings", which extend back from the upright. The base
member is grooved on the bottom to slide along a track, consisting of a length of
$\frac{3}{8}$ inch aluminum square stock. The base is fastened to the foundation plate by
four large Allen head machine screws which travel in two slots provided for easy
movement of the entire tube stand along the track.

Referring back to Figure 1, the diffractometer itself consists of a 15 inch
diameter Bridgeport rotary table, to which a sample holder post, a Geiger Mueller
tube, a receiving slit system, and radiation shielding have been added. The
rotary table provides manual adjustment of the radial angle 2θ with a high degree
of reproducibility. Various sample holders may be placed in the sample holder
post at the center of the table. This is a 1 inch brass post, bored with a $\frac{5}{8}$
inch hole and provided with a clamping collar. There is no provision for a θ
motion of the sample holder.

The primary x-ray beam is stopped by a lead shielding sheet, mounted on
copper for rigidity. The shielding extends slightly over 180°. At the center
of the shielding arc, a jeweler's lathe is mounted, which supports the Geiger
tube and receiving slit assembly. Thus the radial angle may be scanned up to
$2\theta = 90°$ on either side of zero.

B. Slit System

The diffractometer uses the Soller slit optical arrangement of slits. This arrangement is discussed extensively in the book of Klug and Alexander.[1] The design used here is shown in Figure 3, as seen from above. The anode of the x-ray tube is viewed as a vertical line by a Soller slit, a divergence slit, and a scatter slit. The Soller slit, consisting of a series of closely spaced molybdenum sheets, absorbs x-ray photons that are diverging too greatly out of the horizontal plane. In effect, it slices the beam from the vertical line source into a series of nearly parallel beams. The beams recombine to give a vertical line of x-rays with a small vertical divergence (about 2.2°).

The width of the beam is determined by the opening of the divergence slit. Various slits can be used; the present slit gives a beam of 0.2° horizontal divergence.* A scatter slit is placed in front of the divergence slit to pass the primary x-ray beam while absorbing most of the photons scattered by edges of the Soller slit and the divergence slit.

The primary beam strikes the sample at the center of the rotary table and a fraction of the beam is diffracted to various values of the radial angle, 2θ. The intensity at a given angle is measured by a Geiger-Mueller tube after passing through a receiving slit system to reject unwanted photons. The receiving slit, mounted in front of the Geiger tube, is adjusted to a small opening (0.007") to permit good resolution.* The slit is adjustable; both sides move in unison so that the slit width may be varied without changing the location of the center of the slit. In front of the receiving slit, a nickel foil is placed to reject $CuK\beta$ photons while passing $CuK\alpha$ photons. Next, a Soller slit limits the vertical divergence of the incoming photons. Finally, a scatter slit, with an opening of 0.042", rejects photons coming from sources other than the sample. The distance between the receiving slit and the receiving scatter slit may be varied to change the area being viewed. This is adjusted so that the entire irradiated volume of the sample is viewed.

*The horizontal divergence has subsequently been reduced to 0.1°, and the receiving slit opened up to 0.014.

C. <u>Pole Figure Goniometer</u>

The pole figure goniometer is shown in Figure 4. A $\frac{5}{8}$ inch hollow support shaft extends down from the under surface, and fits into the sample holder post of the diffractometer. A second shaft rides inside the hollow shaft, allowing motion of the sample about the vertical axis, the ω motion.* This shaft is driven by a pulse motor through an 18:1 reduction gear train. A microswitch is mounted on the top plate to stop the program at its desired termination point. The microswitch is operated by a striker bar mounted on the sample holder.

The sample is mounted on a holder which allows rotation about the normal of the film sample, the x motion.* The sample mounts on the back face of the holder under a thin aluminum plate, with the axis of the ω rotation passing down the plane of the sample. The holder is driven by a pulse motor mounted on the front side. This pulse motor is geared down by a ratio of approximately 12:1. The ratio is off by 0.1%, since one of the gears is cut with a large prime number of teeth. This is not a serious source of error and is taken into account by the computer program.

The pulse motors, manufactured by the Sigma Instruments Company of South Braintree, Massachusetts, will advance 1/20 of a revolution with every pulse, provided the pulses alternate in polarity. They are discussed further in Section II, part H, <u>Advance Control Unit</u>.

III. Control Unit for New X-Ray Diffractometer

    A. <u>Introduction</u>

The control unit of the diffractometer consists of eight different units, each intended for a separate function and mounted on a separate chassis. These units are:

    1. Offner power supply, type 333

    2. Hamner Timer, model N-803

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*In a previous report the angles ω and x were called φ and ψ respectively. I have changed their names to agree with the accepted terminology.

3. Berkeley scaler, model 2000

4. Baird-Atomic preamplifier, model 231GM

5. Relay power supply

6. Punch control unit

7. Advance control unit

8. Astable multivibrator

Units 1, 2, 3 and 4 are commercial units. Their circuits will not be described here, except where slight modifications have been made. Units 5, 6, 7 and 8 were designed and built at the Polymer Research Institute, and their circuits will be fully described.

B. Offner Power Supply

The Offner power supply provides current at four different potentials: +500 volts,D.C.; +250 volts, D.C.; 6.3 volts,A.C.; and +6.3 volts,D.C. The first three voltages are used in the punch control unit and the advance control unit; the 6.3 volts D.C. is not used.

C. Hamner Timer

The Hamner Timer consists of a six-decade scaler, driven by a 100 cycle signal, which is obtained from a 100 kilocycle crystal oscillator by frequency dividing circuits. The timer is capable of measuring up to 9999.99 seconds, accurate to the nearest hundredth of a second. It was modified in the factory for binary-coded-decimal output. Also available, but not used, are six "staircase" voltage signals which can be read to give the time in decimal form.

Each decade consists of four binary flip-flop circuits, each flip-flop being a 5963 duotriode vacuum tube (see Figure 5). Each flip-flop or "bistable multivibrator", can exist in either of two stable states, in which one triode is conducting and the other one is cut off. (For convenience, these states are referred to as "on" and "off".) The four flip-flop are wired together in such a way that a count of ten is obtained, using a 4-2-2-1 code to store a number. One flip-flop has the value "4" when on, the next has the value "2" when on, etc. Thus the digit "6" is stored as 4 + 2, the first two flip-flops being "on"

and the last two "off". For a given flip-flop, an output signal is obtained which reads +55 volts for "off" and +135 volts for "on". Twenty-four such signals are required to obtain the number stored in the timer.

The timer resets to zero whenever the reset line, which is normally grounded, is opened. The timer is started and stopped by sending external pulses into the appropriate pins on the control jacks. Such pulses should have an amplitude of +20 volts and a rise time of 2 microseconds. The timer can be held in the stopped state by grounding the "hold" line. (The "hold" circuit has been slightly modified from the factory design, which was unreliable.)

The timer also contains a -105 volt bias supply, which is tapped for use in the punch control unit, the advance control unit, and the astable multivibrator.

D. Berkeley Scaler and Baird-Atomic Preamplifier

These two units are used to detect and count the pulses produced by the Geiger-Mueller tube. Both units have standard circuitry which need not be described here, except where modifications have been made.

The scaler contains its own power supply and also provides power for the preamplifier and high voltage for the Geiger-Mueller tube. The scaler has three electronic decades, similar to those in the timer, and an "electronic relay" module. This electronic relay gives a brief 110 volt A.C. signal upon receipt of an input pulse. This input pulse comes from the output of either the second or third decade, depending upon the setting of a manual selector, and thus occurs either at a count of 100 or 1000. The scaler is also provided with a reset button, a start-stop switch, and a mechanical register which is driven by the electronic relay.

Figure 6 shows how the scaler was modified to provide remote control. First, the reset line was brought to a four-pin receptable, leaving the reset button, which is normally closed, in series. The start count switch is to be

left on; counting will start when the reset line is grounded. Next, a one-pin receptable was added to the electronic relay module to provide an output pulse whenever the electronic relay receives an input pulse. This pulse is shaped and attenuated, and then used as the "stop" pulse for the timer.

A small D.C. relay has been added to the scaler, and is driven by the 110 volts A.C. signal from the electronic relay, after appropriate attenuation and rectification. A normally-open pair of contacts from this relay is brought to the four-pin receptacles. The closing of these contacts is used in the relay logic of the punch control unit to signal the end of a count.

E. Relay Power Supply

The circuit diagrams of the relay power supply is shown in Figure 7. The unit is designed to put out five amperes at voltages as high as 60 volts; for its present application the voltage is made continuously variables through use of an autotransformer. The main transformer can be tapped for lower voltages, if desired. The output receptable is not grounded on either terminal, so this unit could be used as a power supply for either positive or negative potentials. In the present apparatus the negative side is grounded in the punch control unit.

F. Punch Control Unit

1. Component parts

The punch control unit, consisting of fifteen relays, a stepping relay, and a vacuum tube, controls the punching of data cards and in addition, starts, stops, and resets the timer and scaler. To accomplish these tasks, it uses four different sources of power: +250 volts, D.C.; +30 volts, D.C.; - 105 volts, D.C. (bias) and 6.3 volts A.C. (filaments).

The fifteen relays, all of which can be operated by 30 volts D.C., fall into two categories according to their resistance. Low resistance relays (200-1000 ohms) are used only with the 30 volt power supply, since they draw too much current for use on the 250 volt supply. High-resistance relays (5000-7000 ohms) are usually used with the 250 volt supply, with an appropriate resistor in series to drop the voltage. The 250 volt supply is called on only when a

special type of action, such as closing after a time delay, is required.

The stepping relay, made by General Electric Co., Ltd. of England, has eight banks of contacts and twenty-five contacts per bank. The wipers are single-ended and adjacent wipers are 180° out of phase, hence at any time, only every other wiper will be wiping a contact. As will be explained later, this feature allows one to use the relay as either a twenty-five-step stepper or as a fifty-step stepper. At present the stepper is wired for twenty-five-step operation; it could easily be changed for a fifty-step operation. The coil of the stepping relay requires one ampere of current at 22 volts; hence it is wired in series with a resistor to operate from the 30 volt supply.

2. Operational logic

The circuits for the Punch Control Unit are shown in Figure 8. In designing the relay circuitry I have chosen to give every relay and every signal a name. The relays are named according to the function they perform; for instance, the "Start Count Relay" (RE2) starts the count cycle. Signals are named for the function they perform or the information they carry, and also bear the appropriate voltage designation. Thus the "250V End of Advance & Punch" signal appears when both advancing and punching are completed. A signal changes its name when it passes through a pair of relay contacts. Two signals, "30V" and "250V" are named only for the voltage level. These signals have not passed through any relay contacts, and subsequently will always be present whenever the "Main Power" toggle switch (SW1) is on.

The program starts when SW1 is turned on. Two signals, "30V" and "250V" appear, and passing through the normally closed contacts of RE1, become the "30V Count" and "250V Count" signals. After a time delay of two seconds the "Start Count" relay (RE2) operates, generating a start pulse for the timer and grounding the reset lines of the scaler and timer. The scaler starts counting and the punch control unit waits for the end of the count (either 100 counts or 1000 counts).

At the end of the count, the scaler's electronic relay operates, instantly stopping the timer and after a small time delay, closing the NO contacts of RE17, mounted within the scaler. The "30V" signal passes through these contacts and becomes the "30V End of Count" signal, which operates RE1, the "Count-Advance Power Relay".

Operation of RE1 removes power from the two "Count" buses and puts power on the two "Advance" buses. The "30V Advance" signal immediately closes RE3, stopping the scaler by opening its reset line, and grounding the timer reset line to prevent the timer from resetting when RE2 drops out. The same signal closes RE25, grounding the timer "hold" line to prevent the timer from accidentally starting. Also, the "30V Advance" signal holds the "Count-Advance Power Relay" closed through the NC contacts of RE4. This is necessary since the "30V End of Count" signal is a momentary signal.

Assuming that SW2 is closed, the "30V Advance" signal will close the "Card Release" relay and after a two-second time delay, the "Punch Start Relay". To understand the punching cycle it is necessary to describe the alterations made within the keyboard of the IBM 026 card punch, shown in Figure 9. The most important line in the keyboard I have called the "Keyboard Common" line. This line is common to all the functions of the keyboard; when it is shorted to the "RELEASE" line, a card is released, when shorted to the "ONE" line a "ONE" is punched, etc. It is necessary to break the short or the function will be repeated over and over. This is accomplished in the keyboard by a coil that I call the "Restorer"; the action of the "Restorer" must be simulated in the punch control unit for successful operation.

A ten-conductor cable connects the card punch with the punch control unit. A relay, RE16, is mounted within the keyboard and operates whenever SW1 and SW2 are closed. Operation of RE16 performs two functions. It disconnects "Keyboard Common" from the keyboard and gives it to the punch control unit, thus

making all the punching keys inoperative. Also, it shorts out the "Auto Feed" switch, thus assuring that a card will be fed when one is released.

Let us return to the discussion of the punching program. When RE13 operates, three functions occur. First, the "Restorer Slave" relay (RE5) is connected with its voltage-dropping resistor in parallel with the "Restorer" coil. Second, the "Release" line is shorted to the "Keyboard Common" line, causing a card to be released and another one fed. Third, voltage is applied to RE15 which will cause it to close after a two-second delay.

When a card is released, the card punch energizes the "Restorer" coil, operating RE5, which in turn operates RE7 and RE8. RE7, the "Release Disconnect Relay" disconnects the release line and is held by power coming through its own NO contacts for the duration of the advance cycle. Diodes are provided to prevent this power from holding RE8.

After the two-second delay, the next card is ready for punching and the "Punch Start Relay", RE15, closes, generating two "Punch Start" signals. At this point, the "Stepper Stopper Relay", RE11, is held by power through one of the stepper wipers, the stepper being in its home position. The "30V Start Punch" signal passes through the NC contacts of RE9 and operates RE10, the "Stepper Starter". This gives 250 volts of power to RE12, the "Relaxation Oscillator Relay". After a time delay, RE12 operates, closing its slave, RE27, which then energizes the stepper coil. When the stepper energizes, its interrupter contacts are opened, removing power from RE12, which drops out after a time delay. The stepper then de-energizes, giving power to RE12 once more. Thus, as long as 250 volts is present at the interrupter, the cycle repeats itself, driving the stepping relay at six cycles per second. This is well within the operating limits of both the stepping relay and the card punch.

Everytime this relay operates, it sends a pulse to the stepper coil and connects the "Keyboard Common" line with either the "0" or the "1" line, and the corresponding digit is punched. Then the "Restorer Slave" receives a pulse, and closes RE8, disconnecting the "Keyboard Common" line to prevent further punching. RE8 is held by power coming through its own NO contacts until the "Relaxation Oscillator" drops out.

The first digit to be punched is selected by the "Motor Selector Slave Relay", RE6, which is controlled from the advance unit. At this point it will suffice to say that this digit tells the computer which angle, χ or ω, is going to be advanced. The remaining twenty-four digits are selected by the "Digit Selector Relay", RE14, and represent the timer reading in a binary decimal code. The operation of RE14 will be described in detail later.

As the stepping relay moves from contact to contact, it passes over one contact to which is wired RE9, the "Stepper Verify Relay". Immediately, the "Stepper Starter" drops out, but the "Stepper Stopper" dropped out earlier and provides voltage for the "Relaxation Oscillator" through its NC contacts. After twenty-five digits are punched the stepper is again at its home position and the "Stepper Stopper" operates, cutting off power from the "Relaxation Oscillator". At this point the two "Punch Start" signals, passing through the NO contacts of RE9 and RE11, become "30V Punch Complete" and "250V Punch Conplete". These signals are carried to receptacles for use in the advance control unit.

At the end of the advance program, the "250V Punch Complete" signal passes through a pair of contacts in the advance control unit and is returned to the punch control unit as the "250V End of Advance" signal. This operates RE4, the "Advance Holding Relay" causing the "Count-Advance Power Relay" to drop out. This ends the advance cycle and starts the count cycle again.

3. Reading of Digits

In order to punch out the reading of the timer it was necessary to convert a voltage level available from the timer to a relay closure. Since the current drain from the timer must be kept small to avoid upsetting the binaries, a vacuum tube

(12AU7) was used to perform this function.

The wiring diagram is shown in Figure 10. The input to the digit selector is through two adjacent wipers of the stepping relay; since they are single-ended, only one will be wiping a contact at a given position. The twenty-four signal lines from the timer are wired to the contact banks. Corresponding contacts on the two banks are cross-wired. No signal is read at the "Home" position, but a wire is connected at this position to the two contact banks for test purposes.

The input comes to the grid of V1, which acts as a cathode follower, reproducing the input voltage at the cathode at a much higher current level. This voltage is bucked against the bias voltage producing a voltage level at the grid of V2. The second triode is either saturated or cut off, depending upon the level of the input voltage. Since the plate resistance includes the coil of RE14, this relay closes when V2 is saturated and opens when V2 is cut off.

The order of the signals on the stepper contacts is such that the six "4" binaries are read first, then six "2"'s, six more "2"'s, and six "1"'s. Therefore, the twenty-four digits can be separated into four six-digit numbers. The time may be obtained in decimal form by multiplying the first number by four, the next two by two, and then summing.

4. Provision for expansion

The punching circuits were designed with the possibility kept in mind that, at some time in the future, it might be desirable to punch out more than the timer reading. With minor alterations, it would be very easy to program the punching of fifty digits instead of twenty-five.

These alterations are shown in Figure 11. First of all, the wire to the "Home" position on bank 7 should be cut. Then this position would no longer be a "Home" position, and the stepper would make fifty steps before stopping. Next, the jumper between wipers 1 and 2 should be cut. Then twenty-five additional inputs could be wired to bank 3.

G. Astable Multivibrator

The purpose of the free-running multivibrator is to generate the pulses required to drive the stepping motors which move the pole figure goniometer. The circuit diagram is given in Figure 12. The circuit consists of a 12AU7 duotriode wired as an astable (free-running) multivibrator.

The 12AU7 exists in two states, in each of which one triode is conducting and the other cut off. To this extent it is similar to the binary flip-flops (bistable multivibrators) used in the timer and elsewhere. The difference is this: in the astable multivibrator neither state is stable, and after a fixed interval the device will spontaneously flip to the other state. Thus, the unit, once started, will flip back and forth from state to state until stopped. The device is stopped by closing the remote on-off contacts, connecting bias voltage through a pad to the grid of V4. When these contacts are shorted, V4 will be cut off and the grid of V3 will decay to ground potential, driving V3 to saturation. This state is stable. When the remote control line is broken, the grid of V4 decays to ground and V4 saturates. The plate of V4 is capacitor coupled to the grid of V3, so this grid is driven negative and V3 cuts off. (This incidentally sends a positive pulse to the grid of V4, which has no effect since V4 is already saturated). The coupling capacitor discharges through the grid resistor of V3, and the grid voltage of V3 decays to ground potential. This causes V3 to saturate again and drives V4 to cut-off. The multivibrator keeps flipping in this manner as long as the control circuit is open. Because of the symmetry of the multivibrator circuit the device spends an equal amount of time in either state. The frequency is dependent upon the values of the coupling capacitors and the grid resistor; the present device operates at about six cycles per second. An output is provided from one of the plates so that the cycles may be counted.

A high-resistance pad in parallel with the plate resistor of V4 gives a voltage signal to a 5696 thyratron tube, which controls the operation of a relay. The cathode of the thyratron is held at B^+ level, so that when V4 saturates, the

voltage signal sends the control grid of the 5696 negative with respect to the cathode (B^+) and the 5000 ohm plate resistor. A diode provides half-wave rectified D.C. for the relay, and a capacitor is wired parallel to the relay to give continuous power.

When the multivibrator gives a voltage signal, the control grid cuts off the thyratron and the relay energizes. When there is no signal, the thyratron conducts and effectively shorts out the relay, so it de-energizes. The contacts of the relay are brought to a twelve-pin receptacle for use in the advance circuits.

The power supply for this unit is contained on the same chassis. The main power transformer provides filament voltages (5 volts and 6.3 volts) and 375 volts AC for B+. The latter is full-wave rectified by a 5U4G tube and smoothed out by an RC filter, giving +375 volts D.C. This voltage is bled to ground through three high-voltage resistors in series. The second resistor is provided with a tap, from which B^+ at the desired voltage (200 volts) may be taken off. A second transformer provides the 60 volt A.C. power for the thyratron. The desired A.C. voltage is taken between the center tap and one leg of the secondary winding; the center tap goes to B^+ to give the desired D.C. level. One leg goes to the thyratron power switch; the other leg is available to power a second such unit.

An octal socket is provided for taking power out of the chassis. Taps are provided for 6.3 volts filament power, ground, +375 volts D.C., B+, and thyratron power (60 volts A.C. with a B+ D.C. level).

H. Advance Control Unit

1. Requirements of the Stepping Motors

The principal purpose of the advance control unit is to control the motions of the pole figure goniometer. This is accomplished by sending the appropriate number of pulses to the appropriate stepping motor.

The wiring of the stepping motors is shown in Figure 13. A given motor is advanced by applying 30 volts of power alternately to each of the two coils in the stepping motor. The motor to get the power is chosen by a relay, RE24, the "Motor Selector Relay". If it is desired to drive both motors simultaneously a jumper can be placed across the two power lines. A resistor and capacitor are provided at all

four coils for spark suppression. The back-and-forth switching of power between the two coils of a given motor is accomplished by the relay in the astable multivibrator unit. Consequently, the motor being driven will step twice for every cycle of the astable multivibrator.

2. Operation of the Advance Control Unit

The circuit diagram of the advance control unit is shown in Figure 14. The advance program starts when the punch control unit sends out the "30V Punch Complete" and "250V Punch Complete" signals. The "30V Punch Complete" signal passes through the NC contact of a microswitch and becomes the "30V Start Motor" signal. This microswitch is mounted on the pole figure goniometer and operates when the entire scan is completed to stop the program.

The "30V Start Motor" signal operates RE21, the "Start Motor Relay". This grounds the advance decade reset line, allowing this unit to count the pulses from the astable multivibrator unit, and also opens the control line of the astable multivibrator. This starts the astable multivibrator and causes the motor selected by RE24 to be driven.

At the end of a prefixed count, the advance decade trips the electronic relay, generating the "30V Stop Motor" pulse. This operates RE22, which stops the astable multivibrator, sends a pulse to the "Advance Stepping Relay", and closes RE19, the "Register Driver Relay". These three coils are held by power through the NO contacts of RE19 until the end of the advance cycle. RE19 also generates the "250V End Punch and Advance" signal which trips either the χ register or ω register, as selected by RE20, and is also sent back to the punch control unit to end the advance cycle.

At the end of the advance cycle RE19, RE21, RE22, and SR2 all lose power. RE22 drops out more slowly than RE21 so that there is no danger of the astable multivibrator starting. At this point, the advance decade resets and the stepping relay wipers are pulled to the next contact. If this brings the stepper to a "Homing"

position, it homes. There is a two-second time delay in the punch control unit
to allow the stepper to home before the timer and scaler start up again.

3. Selection of the Appropriate Stepping Motor.

The purpose of the "Advance Stepping Relay", SR2, is to select which of the
two stepping motors is to be advanced. Whenever the stepping relay is at the "Home"
position the "Motor Selector Relay", RE24 will operate and the ω stepping motor is
selected. At all other positions of the stepping relay, RE24 drops out and the χ
motor is selected.

The advance stepping relay has three banks of contacts, twenty-five con-
tacts per bank, and double-ended wipers. Homing is facilitated by the presence of
a pair of NC contacts which open every time the stepper coil energizes.

When one of these contacts is connected to the stepping relay coil and
the other to 30 volts power, the stepper will step very rapidly, stopping only when
the power is removed. The device is wired so that power is cut off from the inter-
rupter as position 1, the "Home" position.

In actual practice, the stepping relay homes whenever RE23, the "Homing
Relay" is closed. This relay switches the stepper coil from its normal input to the
interrupter contacts. The "Homing Relay", once closed, is held closed by power
through its own NO contacts until RE24, the "Motor Selector Relay", operates. This
relay operates at the home position, causing RE23 to drop out. Two manual buttons
are provided, one to advance the stepper one step, one to cause the stepper to home.

The stepping relay receives a pulse at the end of every advance cycle
unless a toggle switch is opened. Thus the stepping relay counts the number of ad-
vance cycles which have taken place. Jacks are provided which will home the stepping
relay after seven or thirteen advances, thus giving either six or twelve χ advances
for every ω advance. A third jack is not connected to anything. Thus at this jack
there is no homing action and there are twentyfour χ advances for every ω advance.
Additional jacks could be wired up if other programs were desired.

4. Pulse Counting Circuits

The equipment for counting the number of pulses sent to the pulse motor consists of three parts: a one-shot multivibrator for shaping the input pulses, a decade for counting them, and an electronic relay to signal the end of the count. The circuits are shown in Figure 15.

The one-shot multivibrator can exist in two states, but only one is stable. Upon receiving an input pulse it switches to the unstable state, but after a fixed time delay, it reverts spontaneously to the stable state. Its value is due to the fact that it gives a clean, reproducible output pulse for every input pulse capable of triggering it, ignoring the spurious noise which often accompanies the input pulse.

In the stable state, V8 conducts heavily due to its positive grid return. The self-biasing principle is used. Both V7 and V8 have a common cathode resistor, and the grid of V7 is set by a potentiometer at a level such that V7 is cut off whenever V8 conducts, since both cathodes are then at a potential higher than the grid potential of V7. However, when V8 cuts off, the cathode potential drops until V7 conducts.

Negative input pulses are fed to the plate of V7, which is capacitor-coupled to the grid of V8. The grid of V8 is immediately driven in the negative direction, cutting off V8 and causing V7 to conduct. This state is held while the grid of V8 decays up towards + 250 V. Eventually, the grid comes to a high enough voltage and V8 conducts once more, cutting off V7. The output is taken from the plate of V8.

These output pulses are used to drive a decade counter, similar to those used in the timer and the scaler. This unit consists of four binaries wired to give a count of ten. Modifications have been made to allow counts of two or five, providing a little flexibility. The two count is obtained by using the normal input and taking the output from the first binary; the five count is obtained by sending the input to the second binary and using the normal output. (In effect,

one can think of the five count as counting to ten by two's.) Four neon lights
are provided to show which binaries are "on". The input and output are selected
by connecting jumpers to appropriate jacks on the front panel of the advance con-
trol unit.

The output pulse is converted to a relay closure by the electronic relay
unit. Upon receipt of a negative pulse from the decade the "Electronic Relay"
(RE18) closes briefly, then opens again; this in turn closes another relay, RE19,
which is held closed for the duration of the advance cycle.

When there is a steady voltage level at the input of the electronic relay
unit, the grid of V17 is at ground potential and V17 saturates. The grid of V18
is held by a resistor network to a voltage intermediate between the bias voltage
and the plate voltage of V17. With V17 conducting this plate voltage is low enough
that V18 is cut off.

An abrupt negative swing at the input is communicated by an RC network to
the grid of V17, driving it to cutoff. The plate voltage of V17 rises to a value
close to B+, bringing the grid of V18 up with it. This causes V18 to saturate,
closing the relay that is wired to its cathode. The relay is held closed until the
capacitor in the input circuit discharges, bringing the grid of V17 up to ground
potential. Then V17 saturates again, cutting off V18.

IV. Revised Computer Programs

A. Reasons for Changes

In previous reports[2,3,4] computer programs for the IBM 1620 computer were obtained
for correcting the scan data from the x-ray diffractometer for background, absorption,
polarization, and incoherent scattering. The equations and the program appropriate
for a radial x-ray scan were developed by Stein, Powers and Hoshino, while those for
a biaxial scan were developed by the present author. With the construction of the
new diffractometer it became desirable to revise these programs to make them compatible
with the new apparatus. Most of the changes were made in the methods of handling the

data rather than in the calculations themselves. In one instance a limitation on the scope of the programs was removed.

The new diffractometer differs from the old in several ways:

1. There is no crystal monochromator. Soller slits are used instead.

2. The sample holder does not move with changes in the radial angle. In the previous apparatus the sample angle moved an angle 0 when the Bragg angle was changed by 2 0.

3. The radial angle is read in degrees and minutes, rather than in degrees and hundredths.

4. The timer values are punched in a binary decimal code.

These constituted sufficient reason to change the programs. However, the changes were made by making the program more general, rather than writing programs specifically for the new diffractometer. The new programs can handle data from either the old or the new diffractometer.

B. Radial X-Ray Scan Program - Ninth Revision

The latest and perhaps the last revision of the radial scan program was written in Fortran II for the IBM 1620 computer. The source program is listed in table 6. A glossary of the terms used in the program is given in Section VI, part B. Some of the features which distinguish this program from previous versions are the following:

1. The radial angle (2 0) is read in degree and minutes.

2. An option is provided to choose between normal or theta incidence of the x-ray beam on the sample.

3. Sense switches are not used.

4. Data is read in and out of the computer by cards only. The typewriter is used solely for error messages.

5. The time values are read from the cards in a binary decimal code and converted to straight decimal by a subprogram. These times are punched as part of the output record.

If one wants to enter the times in decimal form, this is easily done, as will be explained later.

6. The sample may contain any or all of the following elements: H, C, N, O, F, Cl. The polymer composition is entered on one of the data cards. No other operations are necessary to allow for different polymer compositions.

7. The incoherent scattering factors are found by an interpolation subprogram, not by a table search as before. The interpolation is based on data given in A. H. Compton's book.[6] The interpolation is valid for whatever x-ray wavelength is used. Thus the program can be used for Cu, Mo, Cr or any other x-ray tube simply by reading in the appropriate wavelength in Angstroms.

8. The program does not assume that each data point was obtained by a prefixed count of 1000. The number of counts is entered as part of the data, once for background and separately for the sample.

9. One data card is provided in which one may punch information describing the sample in columns 1-72. This information is included in the output deck. This serves to identify the output deck and prevent mixups.

10. Alphabetical headings are provided so that the output deck, when listed, is self-explanatory. All pertinent input data is included so that such a listing may be reproduced as is in any report, and will give both data and results. (This is a valuable feature, considering the difficulty involved in converting times from BCD to decimal form.)

11. The entire background deck is stored in memory. Background level is recorded every degree: values in between are obtained by interpolation. This should not result in a significant loss in accuracy, but may be circumvented, if desired.

The input data for a sample run is shown in table 7. Complete instructions for the preparation of the input data cards are given in Section VI, Operating Notes, Part C, Preparation of Data Cards. Briefly, the card deck may be described as follows: the first three cards contain data on the sample and the instrumentation. Then the actual scan cards, containing the time values and the 2 θ values, are read. The first such card is obtained at 2 θ = 50° and contains the sample thickness as well. From the intensity at 50°, the level of Compton scattering is determined.

The Compton constant, evaluated at 50°, is used along with the incoherent scattering factor table to evaluate the Compton scattering at any other radial angle.

The subsequent scan cards are read, and the intensities are calculated, corrected for background, absorption, polarization, and incoherent scattering. The end of a scan is indicated when a negative value of the time is read. If a zero value of time is read, the program assumes it to be an error and proceeds to the next card. A typical output deck is listed in table 8.

The computer program must also read the table of incoherent scattering factors and a background scan deck. These cards are kept at the back of the object deck and are read in automatically; when executing the program, one need not worry about them. For this reason they are not considered part of the data deck.

C. Biaxial X-Ray Scan Program - First Revision

The calculations performed by this program are described in a previous report. Briefly, the program handles x-ray data obtained at a fixed radial angle for various orientations of the pole figure goniometer. This data is corrected, as in the radial scan program, for background, absorption, polarization, and incoherent scattering. The resulting intensity distribution gives a full description of the orientation of the crystal plane in question.

The changes made in this program strongly resemble those made in the radial scan program. In fact, this program contains features 1 - 10 listed for the radial scan program. The only feature lacking is number 11. The entire background deck is not stored in memory. Only two background timings are required per scan, and these are read in as part of the initialization data. The two programs were kept compatible, in that whenever the same data was required in both the radial and bi-axial scan programs, the same input format was used. Thus a given card may be used with either program.

There have been several changes in the logic of the program, in addition to those in the input-output procedures. First, the revised program computes the Compton constant for incoherent scattering from data obtained at $2\theta = 50°$. In the

original program the Compton constant was part of the input data; the assumption was that a radial scan would be computed first. This is no longer necessary.

The most drastic change, however, was in the handling of the two orientation angles, χ and ω. In the previous program these angles were punched on each data card. In the present program, one card is read to tell the computer the initial value of each angle, and the amount by which it is incremented, should it be incremented. The first digit on the scan cards, the so-called motor selector digit, tells the computer which angle is incremented. The computer keeps track of both angles in this manner. In addition, the computer keeps both angles in the range between $\pm 360°$ by adding or subtracting 360° when either limit is exceeded.

The source program is shown in table 9, and data for a sample run and the listing of the output are given in tables 10 and 11. For instructions on the preparation of the data cards, see Section V, Operating Notes, Part C, Preparation of Data Cards.

D. Rework Program

Occasionally it is desired to recalculate a few data points within a biaxial scan which has already been calculated. The rework program permits one to do this without running the biaxial scan program, avoiding the cumbersome initialization procedures involved.

The rework program is actually a slight modification of the original biaxial scan program, adapted for the FORGO compiler system. This compiler is a load-and-go system, i.e. the source deck is read and the object program is formed in memory each time the program is ran, and then data cards are read and computations are performed. The object program is not punched out on cards.

A listing of the Rework Program is given in table 12, and sample input and output data are given in tables 13 and 14. Instructions for the preparation of input data cards are given under Operating Notes. The output data cards are in the same format as those from the Biaxial Scan program, except that they contain no card number.

E. Pole Figure Contour Program

The best way to exhibit a pole figure is to plot lines of constant intensity. In this respect, the output of the Biaxial Scan Program is not in its most useful form. The results could be plotted as in Figure 20, with the intensity written in at various points on the pole figure circle. However, the figure shows only a fraction of the data points. If all were shown the graph would be hopelessly filled with numbers. A far better pole figure is shown in Figure 21. The constant intensity contour lines are much more easily interpreted than the numbers on the previous plot.

The purpose of the Pole Figure Contour program is to locate points through which such contour lines can be drawn. It does this by interpolating between the data points produced by the Biaxial Scan Program. The interpolation procedure was modeled after the procedure in a previous report from this laboratory, altered to meet the particular requirements of the x-ray data. The program was written in Kingstran II, an advanced version of Fortran II. The source deck is shown in Table 15.

The first thing the computer does is to generate the contour levels. The levels generated, in counts per minute, are:

100	160	250	400	640
1,000	1,600	2,500	4,000	6,400
10,000	16,000	25,000	40,000	64,000

These levels should completely span any range of intensities to be encountered. The numbers were selected so that each would be greater than the previous by a factor of about 1.6. Unless the program is told otherwise, it will search for contour lines at all these levels. However, one can specify as part of the input data two intensity levels, CONTLO and CONTHI, beyond the range of which no search will be made. If one knows what levels will be encountered this will save computer time.

The computer reads the dimensions of the data arrays. As many as 19 omega levels (0-90° in 5° increments) and 25 chi levels (0-180° in 7.5° increments) may be accommodated. The program assumes that the D_{2h} symmetry exists in the intensity pattern:

1. $I(\omega, \chi) = I(-\omega, \chi)$: plane of symmetry coinciding with the plane of the sample.

2. $I(\omega,\chi) = I(\omega, \chi \pm 180°)$: Twofold symmetry axis coinciding with the film normal direction.

3. $I(\omega,\chi) = I(-\omega, \chi \pm 180°)$: Center of inversion

4. $I(\omega,\chi) = I(\omega,\chi)$: The identity element completes D_{2h}

The entire intensity distribution can thus be described from the distribution in the ranges $0 \leq \omega \leq 90°$ and $0 \leq \chi \leq 180°$. The intensity distribution probably possesses even higher symmetry, possibly complete octahedral symmetry, but this is not assumed.

Certain omega levels are inaccessible, due to blocking by the sample holder. The computer reads these levels on a data card, then generates the omega levels at $5°$ intervals in the range $0 - 90°$, leaving out inaccessible levels. Next, the computer reads correcting factors for certain specified omega levels. These empirical correcting factors, may be used at omega values approaching grazing incidence of the x-rays, where deviation from the theoretical correcting equations may occur. Such factors may be found, as shown in Section V, from scans on unoriented samples.

The program next reads in the biaxial scan cards one at a time. If an empirical correction is to be made, this is done. A corrected version of the scan card is punched out if such correction is made. Next, the data are assigned locations in memory according to the absolute value of omega. The values stored are chi, the count time, and the corrected intensity.

When $\omega = \pm 90°$ is encountered, a special procedure is used. The angle chi is indeterminant at this omega level. (The situation is analagous to trying to define longitude at the north or south pole of the earth). What is done is this: the data from such a card is duplicated to fill in the $\omega = 90°$ level of the matrices. Later the chi values at this level will be arbitrarily assigned chi values coinciding with adjacent values in the $\omega = 85°$ row. Also, the chi values are reduced to the range $0 \leq \chi < 180°$ by adding or subtracting $180°$. The last column of the chi matrix is given values $\chi \leq 180°$ so that chi will increase monotonically at any omega level. Finally, the chi matrix is checked two ways. First, the rows of the

chi matric (i.e. for fixed omega value) are checked to see that chi either increases or decreases monotonically. Next, the columns are checked to see that chi values are nearly equal for consecutive elements in a column. If these conditions are met, the data deck is accepted and the interpolation routines start.

The program takes a contour level and finds all locations where this intensity level occurs. Two different interpolations are done: first, across the rows of the intensity matrix at constant omega values; second, down the columns at nearly constant chi values. The values of omega and chi for the contour level are found by interpolation and punched out. No interpolation is made when the following conditions occur:

(a) The angular span of chi across which the interpolation is made is more than 16°, or

(b) one of the two count times is zero, indicating that the data point is invalid. This indicates that the timer failed to start. Such data points, if they occur, should be rerun before running the contour program.

When searches have been completed for all contour levels, the program types out a message to this effect and then returns to the start of the program, ready to read another input data deck.

Sample input and output listings are given in tables 17 and 18.

V. Calibration and Trial Scans

A. Radial Scans

1. Calibration of the Radial Angle

The main problem in calibrating the diffractometer was setting the zero on the 2θ scale. For this purpose, radial scans were made of powdered samples of lead nitrate.

Samples were prepared by powdering the lead nitrate crystals in a mortar and pestle. A small amount of the powder was smeared on a piece of cellophane tape and covered with a second piece of tape. The resulting sandwich was mounted on a

motor-driven spinning sample holder. In this device, the sample spins about its own normal at a fast rate; the device was used here to average out grain effects. An x-ray photograph showed the sample to be quite grainy, but the spots were sharp, indicating that the lattice had not been deformed by grinding.

The diffractometer was roughly zeroed during the process of alignment (See Section VI, Part A, Alignment of the Diffractometer). A quick scan of the sample revealed strong peaks at 19 1/2°, 22 1/2°, and 32 1/4°. The latter peak was used exclusively since the cellophane tape scatters appreciably at the lower angles.

The peak near 32° was scanned by the prefixed count method on both sides of the diffractometer zero. The scans are shown in Figure 16. The centers of the diffraction peaks were located at +32° 18' and -32° 12'. This indicates that the correct readings should be ± 32° 15'; the zero on the rotary table was adjusted accordingly.

2. Determination of Instrumental Broadening.

The instrumental broadening was estimated by scanning a peak of a lead nitrate sample and assuming that all broadening was due to the instrument. The width at half-height was measured on the curves used for zeroing (Figure 16); the values obtained were 20' and 23'. However, this sample had a high optical density for x-rays (1.41) and a second sample, of optical density 0.26, was prepared to see if the broadening would be affected. The resulting scan is shown in Figure 17. The width at half height was 21', in good agreement with the previous values.

3. Radial Scans of Three Polyethylene Samples

To test the capabilities of the diffractometer, radial scans were made of three different polyethylene samples. The samples had been slightly crosslinked by radiation in a previous experiment (dose = 5 Mr) but were quite crystalline. The thicknesses of the samples were

Sample	Thickness, inches	thickness (μ = 11.3 in^{-1})
1	0.0948	1.072
2	0.0626	0.708
3	0.0363	0.410

Radial scans were made in the manual operating mode (see VI. B. Scanning Procedures), changing the Bragg angle manually between points and using the card punch as the output device. The data deck was run using the radial scan program. The output for sample No. 1 is listed in table 8, and the three corrected intensity curves are shown in figure 18. Three peaks, the 110, 200, and 020 are quite apparent. Three weak peaks, the 210, 120 and 310 are hinted at, but the increment in 2θ was too large to give them shape.

B. Biaxial Scans

1. Test of Absorption Corrections

When one obtains experimental results on biaxial orientation, one must be sure that the effects observed are really orientation effects and not due to varying degrees of absorption by the sample. The effect of sample absorption and of varying scattering volume are taken into account in the biaxial scan computer program. It is necessary to confirm the validity of these corrections.

To do this, biaxial scans were made of three samples not containing any orientation. The samples 1, 2 and 3 are the same ones referred to in the previous section. In terms of absorbing ability these samples are quite thick, and hence would serve as an acid test for the absorption corrections. It is not expected that the normal sample will be this thick, but these experiments established the upper limit on thickness.

The samples were each scanned from $\omega = -70°$ to $\omega = +70°$ using the 110 reflection. The corrected intensity curves are shown in Figure 19. Where the correction is valid, one would expect to find the computed intensity to remain constant. It is evident that the corrections have broken down for sample 1. In sample 2, the correction is valid from $\omega = -60°$ to $+10°$, and in sample 3, from $\omega = -65°$ to $+20°$.

Apparently the assumptions built into the absorption theory do not hold at high sample thickness or at angles approaching grazing incidence. However, this

does not present great difficulties. As long as the sample thickness is 0.036"
or less, the range from $\omega = -65°$ to $\omega = 0°$ is available for use in the transmission
region, for the 110 plane. For other planes, one should not get closer than 10-15°
to grazing incidence. The useable range based on data from an unoriented sample
may be extended by applying empirical corrections*.

2. Sample Pole Figure

A sample pole figure is given in Figure 20. The sample is an experimental
blown polyethylene film provided by the Monsanto Company. The data was obtained
from the 200 reflection in the transmission region. The numbers indicate the relative
density of crystal plane normals at the various orientations. It is seen that the
200 plane normals (i.e. a axes) are concentrated in the plane of the film, near the
transverse direction.

The numbers in brackets were obtained at positive values of the tilt angle
ω, while the others were obtained for negative values. One would expect to find sym-
metry with respect to the plane of the film, and this seems to be the case. The ob-
served symmetry, occurring where the absorption correction varies greatly, confirms
the validity of the absorption correction.

VI. Operating Notes

A. Alignment of the Diffractometer

The following procedure may be used to align the diffractometer:

1. Check the following to see that they are level:

   a. Foundation plate

   b. Top surface of x-ray tube housing

   c. Rotary table

   d. Jeweler's lathe

   e. Both soller slits

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* By reducing the opening of the divergence slit it was found that the useful range
in ω could be expanded, and less deviation from the theoretical absorption correction
occurred.

2. Check to see that the center of the following are all the same distance (10 1/2") above the foundation plate:

    a. Divergence slit

    b. Receiving scatter slit

    c. Receiving slit

    d. Geiger-Mueller tube

3. Level the Geiger-Mueller tube and replace its cover. NEVER APPLY VOLTAGE TO THE GEIGER-MUELLER TUBE WITH THE COVER OFF.

4. Adjust the divergence slit for the desired takeoff angle. The point where takeoff angle is zero is marked. Move the entire divergence soller slit assembly laterally away from this point toward the left to the desired amount. The proper lateral motion ℓ for a takeoff angle θ_{TO} is:

$$\ell = d \tan \theta_{TO}$$

where d = 3 inches is the slit-to-anode distance.

5. Turn the x-ray beam on. BE CAREFUL WITH THE X-RAYS ON. DO NOT PLACE YOUR HANDS IN THE PATH OF THE MAIN X-RAY BEAM. USE THE LEAD BEAM COVER WHEN NECESSARY. Adjust the divergence scatter slit laterally so that the beam is in its center. The opening should be wide enough that the beam does not touch either side of the scatter slit.

6. Place a fluorescent screen at the center of the rotary table and see if the x-ray beam strikes the center. If not, loosen the large Allen screws holding the tube support and move the tube laterally until the beam is centered. Fine adjustment is facilitated by an adjusting screw. Tighten the Allen screws when finished.

7. Remove the Geiger tube assembly and the receiving Soller slit. Move the receiving slit along the lathe until it and the x-ray tube anode are equidistant from the center of the diffractometer. Then adjust the receiving slit for the desired opening, and move the slit laterally until the area viewed by the receiving slit and the receiving scatter slit is centered on the center of the rotary table. This may be easily observed by shining a light through the two slits.

The area viewed should be considerably larger than the area illuminated by the x-ray beam. If not, move the receiving scatter slit along the lathe to increase the area viewed.

8. Replace the receiving Soller slit and Geiger tube, placing Geiger tube as close as possible to receiving slit. Turn on the Geiger tube high voltage. Loosen the two screws to permit lateral motion of the Geiger tube. The Geiger tube can then be adjusted laterally for maximum counting rate. This is best accomplished by placing a sample in the diffractometer, finding a 2θ value where the intensity is strong, then moving the Geiger tube laterally, taking a prefixed count at each position.

9. The pole figure goniometer may be aligned by the following method: first, cover the x-ray port with lead. Place the goniometer in the sample holder shaft and turn the χ angle to approximately 0°. Turn the tilt angle until the sample plane is close to the divergence scatter slit. Then put a flat rectangular sheet in the sample holder, clamping it so its bottom edge hits the two χ = 90° marks and extends close to the scatter slit. An IBM card may be used for this purpose. Level the card by moving the χ angle, and once it is leveled set the indicator at χ = 0. Set the tilt angle protractor to read zero. Move the entire goniometer both vertically and circularly until the IBM cards bottom edge points at the middle of the scatter slit. Then clamp the goniometer in place. Be careful not to overtighten the clamp, since this may bind on the inner shaft and prevent movement.

It is advisable to check the alignment of the goniometer every time a new sample is mounted.

B. <u>Glossary of Terms Used in Computer Programs</u>

1. Biaxial Scan Program

AB: linear absorption coefficient in inches^{-1}. Value is 11.3 for polyethylene.

A,B,C,D: four numbers, six digits each, containing a time value in binary decimal code. If the time is in decimal form, read it as D and set A = B = C = 0.

ANH, ANC, ANN, ANO, ANF, ANCL: the number of atoms of hydrogen, carbon, nitrogen, oxygen, fluorine and chlorine respectively in the repeating unit.

BKNBR: the number of counts (usually 1000) in a background timing.

BT: time, in decimal form, for a background count.

CCO: compton constant; or incoherent scattering constant.

CHIZ: X_o, initial value of angle χ (or ψ).

CINT: corrected x-ray intensity.

COMPT: intensity of Compton scattering at a given 2θ angle.

COS2: $\cos^2 (2\,\theta_M)$. If there is no monochromator crystal, use 1.000.

DCHI: Δ_χ increment in χ (or ψ).

COMG: $\Delta\,\omega$, increment in ω (or ϕ).

FDEG ⎫ radial angle in degrees and minutes. If this angle is in degrees
FMIN ⎭ and tenths, recall that $0.1° = 6'$.

FINCD: digit to indicate type of incidence employed. FINCD = 0. for normal incidence, 1. for theta incidence.

MODE: digit to indicate how the pulse motors are connected. Normal value of MODE is 1. If motor cards are switched, MODE is -1. If both motors run together, MODE is 0.

MTR: Motor selector digit. MTR is 0 for χ advance, 1 for ω advance (as the motors are normally connected).

OMGZ: ω_o, initial value of ω (or ϕ)

PHI: the angle ψ (or ω) in radians.

PHID: same angle in degrees.

PSI: the angle ψ (or χ) in radians.

PSID: same angle in degrees

SCNBR: number of counts (usually 1000 in a sample scan timing.

TEXP: experimental time, in decimal form, for a sample count.

THK: sample thickness in inches

WAVLN: wave length of the x-rays, in Angstroms. Value is 1.5418 for CuK_α

2. Radial Scan Program

Most of the terms used here appear in the Biaxial Scan Program and will not be listed again. Other terms are:

BDEG (I): the ith radial angle, in degrees, at which background is measured.

BT(I): the time obtained for this background count.

BTIME: interplated value of background count time.

SDEG, SMIN: radial angle in degrees and minutes for a sample count. If this angle is in degrees and tenths, recall that $0.1° = 6'$, and punch the proper number of minutes.

3. Rework Program

All of the terms used here appear in the biaxial scan program.

4. Pole Figure Contour Program

CHI(I,J): the i, j value of χ (or ψ)

CHIX: the χ value through which a contour line passes. Associated with corresponding ω value.

CINT(I,J): the i, j value of corrected x-ray intensity, i.e., the value at OMG(I), CHI (I,J)

CONT(K): a level of intensity for which a contour line is desired.

I: index which increases when ω (or ϕ) is increased.

J: index which increases when χ (or ψ) is increased. Returns to 1 when ω is increased.

K: index for the various contour levels

NCHI: the $\chi:\omega$ ratio, i.e. the number of χ increments for each ω increment. NCHI is the maximum value of the index J.

NOMG: number of ω values. NOMG is the maximum value of the index I.

OMG(I): the ith value of ω (or ϕ)

OMGX: the ω value through which a contour line passes. Associated with a corresponding χ value.

TEXP(I,J): the experimental count time for the i, j data point.

TILT(M): an omega value at which the empirical correction factor x (M) is to be used.

TRIP(NBR): an omega value which is experimentally inaccessible and is to be skipped.

X (M): an empirical correction factor which corrects deviation from the theoretical absorption at ω = TILT(M). Based on data from an ω scan of an unoriented sample.

C. Preparation of Data Cards

The formats for the data cards for the four programs are shown in Tables 1, 2, 3, and 4. Several points are worth mentioning:

1. Any data item which is zero may be left blank.

2. Punches in fields which are not to be read will cause no trouble.

3. Comments may be punched on any card after column 44. These will not be read.

4. In the Radial and Biaxial Scan programs, the incoherent scattering factor table is at the end of the object deck and are not considered part of the data deck. Likewise, the background deck is part of the object deck in the Radial Scan program. For this reason, do not use this instruction "4900409" to restart these programs. The machine will try to read this data again if this is done.

5. Any number of data decks may be processed, provided each deck has a proper "Last" card (does not apply for the Pole Figure Contour Program).

6. The output decks are self-explanatory.

D. Scanning Procedures

1. Radial Scans

At present, the radial angle is not automated, but scans may be done on a semiautomatic basis if desired. The procedure is as follows:

a. Mount the sample at normal incidence

b. Turn off the Astable Multivibrator.

c. Turn off the main switch on the punch control unit; turn on the automatic punching switch.

d. Place cards in the card punch hopper, then press the feed key twice. A card should be registered. Punch identifying remarks on this card, but do not release it.

e. Manually adjust the radial angle to 50°. Turn the main switch on. The count cycle should start. At the end of the count, the time will be punched in columns 1-25.

f. Turn the main switch off. Punch SDEG, SMIN and THK in the appropriate fields, using decimal points.

g. Repeat at other desired angles (THK need not be punched again.) Angles may be taken in any order need not be regularly spaced. Any value within the range of the background deck may be used. To avoid backlash error, always approach an angle with a clockwise motion of the worm gear.

h. After the last scan card, punch a card with -1 in the field 2-7 to end the scan.

i. Place appropriate cards for cards 2 and 3 at the front of the deck.

2. Biaxial Scans

a. Startup

Starting biaxial scan is a complicated procedure, but once started, an enormous amount of data can be obtained without further intervention. The procedure is complicated by the problem of starting the pulse motors at the right position. Each motor consists of two coils, one driven by an N.C. contact and one by an N.O. contact of the Astable Multivibrator relay. At any given motor position, only one of the two coils can cause the shaft to turn. We must start the motors in positions such that the N.O. coil can turn the shaft when the first pulse is received; otherwise a pulse will be missed. This is accomplished by positioning the motors with current passing through the N.C. coils.

The procedure is as follows

1) Turn off the Astable Multivibrator. Check the advance control unit to see that the χ pilot light is on. If not, push the step button.

2) Check to see if the striker bar is in the right position. Move the ω drive by hand to a point slightly past the desired stopping point. Adjust the microswitch so it is just about to operate. Then back off from the microswitch.

3) Manually adjust the χ drive to the desired starting value, and loosen the set screw on the driving gear. It may be necessary to disengage and re-engage the gear to get the screw properly oriented.

4) Turn on the main switch and the automatic punch switch on the punch control unit. When the count cycle starts, depress the stop count switch. The device will go through the punch cycle and stop at the beginning of the advance cycle, with power on the N.C. coil of the χ stepping motor. The "χ pulses" pilot light will go on.

5) Hold the χ angle at the desired position and tighten the set screws.

6) Loosen the set screw on large gear surrounding the brass goniometer support shaft. Move the goniometer to the desired starting position. Push the homing button. The "ω pulses" light will go on.

7) Power is now on the ω motor. Tighten the set screw. The motors are in the desired positions. Turn the main switch on punch control off, and turn on the Astable Multivibrator.

8) Place blank cards in the card hopper. Turn Auto Feed on, punch release twice. Manually punch a "No. 1" and a "No. 7" card, as in Table 2 . Do not release the "No. 7" card.

9) Check the control settings:

a) Zero the χ, ω, and scaler registers.

b) Check the increment on the advance decade.

c) Check the $\chi:ω$ ratio value

d) Check the "Double program-single program" switch

e) Check the status of the advance stepping relay. Normally, this should be one step away from the "Home" position at the beginning of a scan.

f) Check the radial angle

g) Remove the lead beam stop

h) Check the timer on the x-ray generator, and the k_v and m_a values.

10) Start the program by turning the main switch on.

11) At the end add cards 2-6 and a "Last" card to the data deck.

b. Suggested Scanning Programs

Some possible scan programs are indicated in Table 5. The simplest programs are the χ scan and the ω scan, each being useful when complete symmetry exists with respect to the other angles. A fine increment is desirable here and increments of 2° in ω and 3° in χ are available.

In the biaxial scans, one can set the decade for either a coarse or a fine scan. The latter will give four times as many data points in a given area of the pole figure. The change in χ between ω advances is an important figure. If this is 360°, an entire pole figure is produced. If it is 180°, half the pole figure is produced, but the other half is easily obtained by assuming a center of symmetry exists. If it is 90°, one-fourth of the pole figure is produced. The entire pole figure can be obtained if one knows a line of symmetry as well as the center of symmetry. If the assumed line of symmetry is off, due to inaccurate mounting of the sample, the pole figure will be in error. For this reason a value of 180° is preferred. Using a value of 360° is a duplication of effort, and is not preferred.

VII. References

1. H. P. Klug and L. E. Alexander, X-Ray Diffraction Procedures, John Wiley, New York, (1954), Chap. 11

2. R. S. Stein, J. Powers and S. Hoshino, ONR Technical Report No. 33, Project No. 356-378, Contract No. 3357(00), University of Massachusetts, July 7, 1961

3. C. R. Desper, ONR Technical Report No. 33a, Project NR356-378, Contract Nonr 3357(01) University of Massachusetts, July 24, 1963.

4. C. R. Desper, ONR Technical Report No. 51, Project NR 356-378, Contract Nonr 3357(01), University of Massachusetts, September 10, 1962.

5. D. G. LeGrand, D. Keedy and R. S. Stein, ONR Technical Report No. 26, Project NR 356-378, Contract Nonr 2151(00), University of Massachusetts, December 2, 1960.

TABLE I

Format for Data Cards, Radial X-Ray Scan

Field No.	1	2	3	4	5	6	7	8	Comments
Columns	1	2-7	8-13	14-19	20-25	26-31	32-37	38-43	
1	Remarks identifying the sample may be punched in columns 1-72								
2	-	ANH	ANC	ANN	ANO	ANF	ANCL	-	Cards 2 and 3 are the
3	-	FINCD	AB	COS2	WAVLN	BKNBR	SCNBR	-	same as in Biaxial Program
4	-	A	B	C	D	50.	0.	THK	Sample at 50°
5-ff	-	A	B	C	D	SDEG	SMIN	-	Scan cards
Last	-	-1	-	-	-	-	-	-	

See Part III, Revised Computer Programs, for a glossary of terms.

TABLE II

Format for Data Cards, Biaxial X-Ray Scan

Field No.	1	2	3	4	5	6	7	8	Comments
Columns	1	2-7	8-13	14-19	20-25	26-31	32-37	38-41	
1	Remarks identifying the samples may be punched in columns 1-72								
2	-	ANH	ANC	ANN	ANO	ANF	ANCL	-	
3	-	FINCD	AF	COS2	WAVLN	BKNBR	SCNBR	-	
4	-	A	B	C	D	-	-	-	Background at 50° 2 Θ
5	-	A	B	C	D	50.	0.	THK	Sample at 50° 2 Θ
6	-	A	B	C	D	-	-	-	Background at Bragg Angle
7	-	OMGZ	CHIZ	DOMG	DCHI	FDEG	FMIN	MODE	
8-ff	MTR	A	B	C	D	-	-	-	Scan cards
Last	-	-1	-	-	-	-	-	-	

See Part III, Revised Computer Programs, for a glossary of terms.

TABLE III

Format for Data Cards, Rework Program

Field No.	1	2	3	4	5	6	Comments
Columns	1	2-7	8-13	14-19	20-25	26-31	
1	-	-	AB	COS2	BKNBR	SCNBR	Same as card 2, Biaxial Program
2	-	FDEG	FMIN	BT	COMPT	THK	
3-ff	-	PHID	PSID	TEXP	-	-	
Last	-	0.	0.	0.	-	-	

TABLE IV

Format for Data Cards, Pole Figure Contour Program

Field No.	1	2	3	4	5	Comments
Columns	1	2-7	8-13	14-19	20-25	
1						Remarks identifying the sample may be punched in columns 1-72
2	-	FNOMG	FNCHI	CONTLO	CONTHI	
3	-	TRIP(1)	TRIP(2) etc.	...		As many as 12 of these
4	-	TILT(1)	X(1)	TILT(2)	X(2)	As many as 6 each of these
5-ff						Output Cards from Biaxial Scan or Rework Programs

Note: The order of the Biaxial Scan output cards is important

1. Within a given Omega level, the Chi values should increase monotonically.

2. The different Omega levels may be in any order.

3. The level Omega = $\pm$ 90° should be represented by only one card. The Chi value on that card is immaterial.

TABLE V

Suggested Automatic Scans

Scan Program	Double or Single	χ:ω ratio	Decade	Δω	Δχ *	Change in χ between ω advances
Fine Biaxial Scan	Double	24:1#	5	5°	7.5°	180°
		12:1				90°
Coarse Biaxial Scan	Double	24:1	10	10°	15°	360°
		12:1#				180°
		6:1				90°
χ Scan	Single	-	10	-	15°	-
			5	-	7.5°	-
			2#	-	3°#	-
ω Scan	Single	-	10	10°	-	-
			5	5°	-	-
			2#	2°#	-	-

*Nominal increment in χ. The actual increment is 0.1% greater. The computer program takes this into account.

#Preferred Programs.

TABLE 6. LISTING OF RADIAL XRAY SCAN PROGRAM

```
C         RADIAL XRAY SCAN PROGRAM              PROBLEM 196    DICK DESPER
C         NINTH REVISION - 7/30/64 - FORTRAN II
C         FOR RADIAL SCANS WITH NEW DIFFRACTOMETER
C         BRAGG ANGLE SPECIFIED IN DEGREES AND MINUTES
C         OPTION FOR NORMAL OR THETA INCIDENCE
C         INPUT AND OUTPUT ON PUNCHED CARDS - SENSE SWITCHES NOT USED
C         PROVISION FOR SAMPLES CONTAINING H.C.N.O.F.CL
          DIMENSION BT(361).BDEG(361)
          DIMENSION Y(7.6).ANS(6)
          COMMON Y.X.ANS.STRFH.STRFC.STRFN.STRFO.STRFF.STRFCL
          READ 600.((Y(I.J).I=1.7).J=1.6)
          J=0
  2       DO 6 I=1.361
          READ 42.A.B.C.D.BDEG(I)
          BT(I)=DECML(A.B.C.D)
          IF (BT(I)) 200.980.6
  6       CONTINUE
  5       PRINT 52
          STOP
  980     PRINT 85
          STOP
C         VERIFICATION OF BACKGROUND DECK
  200     N=I-1
          PRINT 45
          DO 201 I=2.N
          TEST=BDEG(I)-BDEG(I-1)
          IF (TEST) 202.203.201
  201     CONTINUE
          GO TO 990
  202     PRINT 53.BDEG(I-1)
          GO TO 204
  203     PRINT 54.BDEG(I)
  204     PRINT 55
          STOP
  100     PRINT 86.J
          PAUSE
  990     READ 87
          READ 40.ANH.ANC.ANN.ANO.ANF.ANCL
          SUM=ANH+ANC+ANN+ANO+ANF+ANCL
          READ 41.FINCD.AB.COS2.WAVLN.BKNBR.SCNBR
          INCD=FINCD
  4       CNT=0.
          IZERO=N
          J=J+1
          PUNCH 48.J
          PUNCH 87
          PRINT 48.J
          PRINT 87
  20      CONTINUE
  10      READ 44.A.B.C.D.SDEG.SMIN.THK
          TEXP=DECML(A.B.C.D)
  12      IF (TEXP) 100.205.1
C         TEXP = MINUS INDICATES END OF SCAN. TEXP = ZERO TREATED AS A MISTAKE
  205     PUNCH 63.SDEG.SMIN.TEXP
          IF (CNT) 13.13.20
  1       DO 15 I=IZERO.N
          TEST=SDEG-BDEG(I)
          IF (TEST) 15.18.15
```

```
      15     CONTINUE
             DO 981 I=1,IZERO
             TEST=SDEG-BDEG(I)
             IF (TEST) 981,18,981
     981     CONTINUE
C            IF THIS POINT IS REACHED, THERE IS NO MATCH IN BACKGROUND DECK
      19     PUNCH 51,SDEG
             PRINT 51,SDEG
             IF (CNT) 13,13,20
      13     PUNCH 49
             PRINT 49
C            NO MATCH FOR FIRST SCAN DATA CARD, HENCE CCO CANNOT BE CALCULATED
C            COMPUTER ABORTS THIS SCAN - LOOKS FOR ANOTHER SCAN TO CALCULATE.
       9     READ 44,A,B,C,D,SDEG,SMIN,THK
             TEXP=DECML(A,B,C,D)
             IF (TEXP) 100,9,9
C            MATCH FOUND IN BACKGROUND DECK
      18     THET=3.1415927*((SDEG+SMIN/60.)/360.)
             IZERO=I
             BTIME=BT(I)
             IF (SMIN) 17,8,17
      17     DTHET=60.*(BDEG(I+1)-BDEG(I))
             DBT=BT(I+1)-BT(I)
             BTIME=BTIME+DBT*SMIN/DTHET
       8     COS2T=COSF(2.*THET)
             COST=ABSF(COSF(THET))
             SINT=SQRTF(1.-COST*COST)
             X=SINT/WAVLN
             CALL INTER
             STRF=ANH*STRFH+ANC*STRFC+ANN*STRFN+ANO*STRFO+ANF*STRFF+ANCL*STRFCL
             STRF=STRF/SUM
             IF (CNT) 986,986,987
     986     ABTH=AB*THK
C            OPTION BETWEEN NORMAL OR THETA INCIDENCE
     987     IF (INCD) 23,22,23
C            NORMAL INCIDENCE CASE   USE FINCD=ZERO
      22     ARG=ABTH*(1./COS2T-1.)
             IF (ABTH) 910,911,910
     911     CABS=1.
             GO TO 24
C            TEST FOR FORWARD OR BACK REFLECTION CASES (NORMAL INCIDENCE)
     910     IF (COS2T) 27,27,28
      27     ARG=-1.*ARG
      28     CABS=ARG/(1.-EXPF(-1.*ARG))
             GO TO 24
C            THETA INCIDENCE CASE   USE FINCD=1.
      23     CABS=COST*EXPF(ABTH/COST)
      24     CPOL=(1.+COS2)/(1.+COS2*COS2T*COS2T)
             DIF=60.*(SCNBR/TEXP-BKNBR/BTIME)
             COF=DIF*CPOL*CABS
             IF (CNT) 25,25,26
C            CALCULATION OF INCOHERENT SCATTERING CONSTANT
      25     CCO=COF/STRF
             NDEG=SDEG
             PUNCH 67,THK
             PUNCH 46,CCO,NDEG
             PUNCH 65,BTIME,TEXP
             PUNCH 50
             CNT=1.
             GO TO 20
```

```
C         CALCULATION OF CORRECTED INTENSITY
  26      CINT=COF-STRF*CCO
          PUNCH 47,SDEG,SMIN,TEXP,CINT
          GO TO 20
  602 CONTINUE
  40      FORMAT (1X,6F6.0)
  41      FORMAT (1X,4F6.4,2F6.0)
  42      FORMAT (1X,4F6.2,F6.1)
  43      FORMAT (1X,F6.2,F6.1)
  44      FORMAT (1X,4F6.2,2F6.1,F6.4)
  45      FORMAT (42H BACKGROUND AND SCATTERING FACTORS LOADED /
      117H READ SCAN DECKS )
  46      FORMAT (5H CCO=F10.4,14H EVALUATED AT 14,5H DEG )
  47      FORMAT (1X,2F6.1,F8.2,F9.2)
  48      FORMAT (36H XRAY RADIAL SCAN            SCAN NO. I4)
  49      FORMAT (30H THIS SCAN CANNOT BE COMPUTED )
  50      FORMAT (31H    DEG    MIN    TIME    INTENSITY )
  51      FORMAT (1X,F6.1,25X,32H DEGREES NO MATCH IN BACKGROUND    )
  52      FORMAT (39H BACKGROUND DECK EXCEEDS STORAGE TABLE )
  53      FORMAT (41H BACKGROUND DECK CARD OUT OF SEQUENCE AT F6.1,5H DEG )
  54      FORMAT (32H BACKGROUND DECK DUPLICATION AT F6.1,5H DEG )
  55      FORMAT (53H PLEASE RECTIFY ERROR AND BRANCH TO START OF PROGRAM )
  53      FORMAT (1X,2F6.1,F8.2,11X,23H OMITTED AS IMPOSSIBLE )
  65      FORMAT (17H DATA USED - BKG F8.2,13H SEC. SAMPLE F8.2,5H SEC )
  67      FORMAT (18H SAMPLE THICKNESS F6.4,8H INCHES )
  85      FORMAT (36H BACKGROUND CARD CONTAINS TIME=ZERO )
  86      FORMAT (20H END OF RADIAL SCAN I4/33H PUSH START TO READ ANOTHER S
      1CAN )
  87      FORMAT (72H
      1                                  )
  600 FORMAT(10X,7F5.0)
          END
```

TABLE 7

EXAMPLE OF RADIAL SCAN INPUT DATA DECK

```
RADIAL SCAN CROSS-LINKED POLYETHYLENE SAMPLE NO. 1    10/27/64
   4.      2.
   0.      11.3   1.00  1.5418  1000.  1000.
000001100111100111100111 50.     0.
000010000010100100100011 10.     0.
000010000010000101000000 11.     0.
...etc.
000011100111100111000C0110 34.   0.
000000100111100111000011 35.     0.
000001000101000111100011 35.    10.
000001000111000111000001 35.    20.
000001100101100111100011 35.    30.
000000000010000010110000010 35.  40.
000000000001010011010010011 35.  50.
000010000010000111100011 36.     0.
000010000010000100100001 36.    10.
000000000000110010110010001 36.  20.
000000000001110011110010010 36.  30.
000010100010100110000110 36.    40.
000010100011100111100111 36.    50.
000000000010100010110000010 37.  0.
000000000100100111100001 37.    10.
000000000100000110100011 37.    20.
000000100110100110100011 37.    30.
000010000011100111100110 38.     0.
000000100011100111100110 39.     0.
000010100011100101000011 40.     0.
0-10000000000000000000000  0.    0.
```

TABLE 8

TYPICAL OUTPUT DATA, RADIAL SCAN PROGRAM

```
XRAY RADIAL SCAN              SCAN NO.   1
RADIAL SCAN  CROSS-LINKED POLYETHYLENE SAMPLE NO. 1  10/27/64
SAMPLE THICKNESS  .948 INCHES
CCO-  975.2818 EVALUATED AT   50 DEG
DATA USED - BKG   999.25 SEC. SAMPLE    55.99 SEC
  DEG   MIN   TIME   INTENSITY
  20.0  10.0   7.71   7405.18
  20.0  20.0   7.23   7985.49
  20.0  30.0   7.10   8164.84
  20.0  40.0   6.51   9023.87
  20.0  50.0   5.55  10803.48
  21.0   0.0   4.45  13785.57
  21.0  10.0   2.99  21122.38
  21.0  20.0   2.23  28768.47
  21.0  30.0   2.20  29221.35
  21.0  40.0   2.82  22565.89
  21.0  50.0   4.51  13675.04
  ...etc.
  35.0  20.0  44.91    147.09
  35.0  30.0  43.99    186.44
  35.0  40.0  40.32    356.88
  35.0  50.0  34.15    725.74
  36.0   0.0  29.33   1124.15
  36.0  10.0  26.13   1470.81
  36.0  20.0  30.45   1026.43
  36.0  30.0  34.54    707.04
  36.0  40.0  39.16    425.06
  36.0  50.0  39.58    403.52
  37.0   0.0  40.52    355.57
  37.0  10.0  42.25    273.38
  37.0  20.0  43.12    235.57
  37.0  30.0  44.18    190.90
  38.0   0.0  39.44    429.47
  39.0   0.0  35.48    691.19
  40.0   0.0  27.56   1434.91
```

TABLE 9. LISTING OF BIAXIAL XRAY SCAN PROGRAM

```
C       BIAXIAL XRAY SCAN PROGRAM        PROBLEM 196   DICK DESPER
C       FIRST REVISION - 8/7/64 - FORTRAN II
        DIMENSION Y(7,6),ANS(6)
        COMMON Y,X,ANS,STRFH,STRFC,STRFN,STRFO,STRFF,STRFCL
        READ 600,((Y(I,J),I=1,7),J=1,6)
        TYPE 601
        J=0
 100    J=J+1
        K=0
        READ 54
        READ 41,ANH,ANC,ANN,ANO,ANF,ANCL
        SUM=ANH+ANC+ANN+ANO+ANF+ANCL
        READ 42,FINCD,AB,COS2,WAVLN,BKNBR,SCNBR
        INCD=FINCD
C       READIN OF DATA FOR COMPUTATION OF INCOHERENT SCATTERING
        READ 44,A,B,C,D
        BT=DECML(A,B,C,D)
        READ 44,A,B,C,D,FDEG,FMIN,THK
        TEXP=DECML(A,B,C,D)
        GO TO 103
C       READIN OF INITIAL DATA FOR SCAN
 102    READ 44,A,B,C,D
        BT=DECML(A,B,C,D)
 90     READ 43,OMGZ,CHIZ,DOMG,DCHI,FDEG,FMIN,MODE
        KARD=1
        DCHI=1.001*DCHI
C       MODE = 1    PULSE MOTOR CABLES CONNECTED AS LABELLED
C       MODE = -1   CABLES REVERSED (CHI CABLE TO OMEGA MOTOR , ETC)
C       MODE = 0    JUMPER INSTALLED SO MOTORS RUN SIMULTANEOUSLY
 103    ABTH=AB*THK
        NDEG=FDEG
        NMIN=FMIN
        THETD=0.5*(FDEG+FMIN/60.)
        THET=RADIAN(THETD)
        COS2T=COSF(2.*THET)
        CPOL=(1.+COS2)/(1.+COS2*COS2T*COS2T)
        COST=ABSF(COSF(THET))
        SINT=SQRTF(1.-COST*COST)
        X=SINT/WAVLN
        CALL INTER
        STRF=ANH*STRFH+ANC*STRFC+ANN*STRFN+ANO*STRFO+ANF*STRFF+ANCL*STRFCL
        STRF=STRF/SUM
        IF (K) 116,116,101
C       COMPUTATION OF INCOHERENT SCATTERING CONSTANT
 116    K=1
        DIF=60.*(SCNBR/TEXP-BKNBR/BT)
C       OPTION BETWEEN NORMAL OR THETA INCIDENCE
        IF (INCD) 108,105,108
C       NORMAL INCIDENCE CASE - USE INCD = ZERO
 105    ARG=ABTH*(1./COS2T-1.)
        IF (ARG) 505,506,505
 506    CABS=1.
        GO TO 111
 505    IF (COS2T) 109,109,110
 109    ARG=-1.*ARG
 110    CABS=ARG/(1.-EXPF(-1.*ARG))
        GO TO 111
C       THETA INCIDENCE CASE   USE INCD = ONE
```

```
108    CABS=COST*EXPF(ABTH/COST)
111    CCO=DIF*CABS*CPOL/STRF
       PUNCH 60,J
       TYPE 60,J
       PUNCH 54
       TYPE 54
       IF (CCO) 501,502,502
501    PUNCH 50
       TYPE 50
       STOP
502    PUNCH 64,CCO,NDEG,NMIN
       PUNCH 65,BT,TEXP
       PUNCH 67,THK
       GO TO 102
101    PHID=OMGZ
       PSID=CHIZ
       COMPT=CCO*STRF
       PUNCH 66,NDEG,NMIN,BT
       PUNCH 68,COMPT
       PUNCH 61
       PUNCH 62
C      READIN OF SCAN CARDS
106    READ 47,MTR,A,B,C,D
       TEXP=DECML(A,B,C,D)
C      TEXP = MINUS INDICATES END OF SCAN, TEXP = ZERO TREATED AS A MISTAKE
107    IF (TEXP) 500,12,5
500    PUNCH 55,J
       TYPE 55,J
       GO TO 100
5      PSI=RADIAN(PSID)
       PHI=RADIAN(PHID)
19     COSA=SINF(PSI)*COSF(PHI)
       RAD=SQRTF((1.0-ABSF(COSA))/(1.0+ABSF(COSA)))
C      TEST FOR TILT
       IF (PHID) 6,7,6
6      TEST=180.0-ABSF(PHID)
       IF (TEST) 11,7,11
11     TEST=360.0-ABSF(PHID)
       IF (TEST) 15,7,15
C      TESTS FOR REFLECTION OR TRANSMISSION
15     COSP=COSF(THET+PHI)
       COSM=COSF(THET-PHI)
       TEST=COSP*COSM
       IF (TEST) 16,12,13
12     PUNCH 63,PHID,PSID,TEXP
       GO TO 23
C      UNTILTED SAMPLE
7      CABS=COST*EXPF(ABTH/COST)
       TEST=COSF(PSI)*COSF(PHI)
       IF (TEST) 17,4,18
17     COSD=-1.0
       GO TO 8
4      COSD=0.0
       GO TO 8
18     COSD=1.0
       GO TO 8
C      TRANSMISSION METHOD
13     SECP=1.0/ABSF(COSP)
       SECM=1.0/ABSF(COSM)
       SECD=SECP-SECM
```

```
          CABS=(ABTH*SECD)/(SECM*(EXPF(-ABTH*SECM)-EXPF(-ABTH*SECP)))
          GO TO 14
C         REFLECTION METHOD
   16     SECP=1.0/ABSF(COSP)
          SECM=1.0/ABSF(COSM)
          SECS=SECP+SECM
          CABS=(ABTH*SECS)/(SECM*(1.0-EXPF(-ABTH*SECS)))
   14     SINA=SQRTF(1.0-COSA*COSA)
          IF (SINA) 2,4,2
    2     COSD=COSF(PSI)*COSF(PHI)/SINA
C         COMPUTATION OF CORRECTED INTENSITY AND PUNCHING OF RESULTS
    8     DIF=60.*(SCNBR/TEXP-BKNBR/BT)
          CINT=DIF*CABS*CPOL-COMPT
          PUNCH 49,PHID,PSID,TEXP,COSD,COSA,RAD,CINT,KARD
          IF (CINT) 503,504,504
  503     TYPE 51,KARD
  504     KARD=KARD+1
C         INCREMENTING THE SAMPLE ANGLES
   23     TEST=MODE*(2*MTR-1)
          IF (TEST) 22,24,21
   21     PHID=PHID+DOMG
          GO TO 104
   24     PHID=PHID+DOMG
   22     PSID=PSID+DCHI
C         REDUCTION OF ANGLES TO RANGE -360 TO +360 DEGREES
  104     IF (PHID) 112,113,114
  112     TEST=PHID+360.
          IF (TEST) 117,117,113
  117     PHID=TEST
          GO TO 112
  114     TEST=PHID-360.
          IF (TEST) 113,115,115
  115     PHID=TEST
          GO TO 114
  113     IF (PSID) 118,106,119
  118     TEST=PSID+360.
          IF (TEST) 120,120,106
  120     PSID=TEST
          GO TO 118
  119     TEST=PSID-360.
          IF (TEST) 106,121,121
  121     PSID=TEST
          GO TO 119
   69     CONTINUE
   41     FORMAT (1X,6F6.0)
   42     FORMAT (1X,4F6.4,2F6.0)
   43     FORMAT (1X,6F6.1,I3)
   44     FORMAT (1X,4F6.2,2F6.1,F6.4)
   47     FORMAT (I1,4F6.2)
   49     FORMAT (1X,2F6.1,F8.2,3F8.5,F9.2,13X,I5)
   50     FORMAT (15H DATA IN ERROR /26H CCO FOUND TO BE NEGATIVE )
   51     FORMAT (35H NEGATIVE INTENSITY AT OUTPUT CARD I4)
   54     FORMAT (79H
        1                                         )
   55     FORMAT (55X,22H END OF BIAX SCAN NO. I3)
   60     FORMAT (25X,28H BIAXIAL XRAY SCAN - NUMBER I3)
   61     FORMAT (54H OMEGA  CHI   TIME    COSINE   COSINE   RADIUS INTENSITY)
   62     FORMAT (53H -PHI- -PSI-   SEC    DELTA    ALPHA           CTS/MIN/)
   63     FORMAT (1X,2F6.1,F8.2,34X,23H OMITTED AS IMPOSSIBLE )
   64     FORMAT (5H CCO=F10.4,14H EVALUATED AT I4,5H DEG I4,5H MIN )
   55     FORMAT (17H DATA USED - BKG F8.2,13H SEC, SAMPLE F8.2,5H SEC )
```

```
66      FORMAT (12H     2THETA =14.5H DEG 14.13H MIN     BKG =F8.2.5H SEC )
67      FORMAT (18H SAMPLE THICKNESS F6.4.8H INCHES )
68      FORMAT (33H INTENSITY OF COMPTON SCATTERING F9.2.9H CTS/MIN  /)
600     FORMAT(10X.7F5.0)
601     FORMAT (27H BIAXIAL XRAY SCAN PROGRAM /38H INCOHERENT SCATTERING F
     1ACTORS LOADED   )
        END
```

TABLE 10

TYPICAL INPUT DATA, BIAXIAL SCAN PROGRAM


```
SAMPLE 32A   110 PLANE   REFLECTION REGION   DATE OCTOBER 22, 1964
4.     2.    0.    0.    0.    0.
0.     11.3  1.00  1.5418 1000. 1000.
001110001110101111011101 50.          FIFTY DEGREE BACKGROUND CARD
100110000111000111101l010 50.  0.    .0096 FIFTY DEGREE CARD SAMPLE 32A
000111000111001110011001 21.  20.    BACKGROUND COUNT 21 DEG 20 MIN
85.  0.   5.   7.5   21.  20.   1    SAMP 32A   110 PLANE   REFLECTION RGN
000000000011000011l000110
000001100011100010l000101
...etc.
000000000010100010l000110
000001000011100010l000101
...etc.
100000000000000011l0000111
-1
```


TABLE 11

TYPICAL OUTPUT, BIAXIAL SCAN PROGRAM


```
                 BIAXIAL XRAY SCAN - NUMBER   1
SAMPLE 32A    110 PLANE    REFLECTION REGION    DATE OCTOBER 22, 1964
CCO=  181.3172 EVALUATED AT    50 DEG    0 MIN
DATA USED - BKG   999.25 SEC, SAMPLE   198.52 SEC
SAMPLE THICKNESS  .0096 INCHES
   2THETA =  21 DEG   20 MIN    BKG =  398.61 SEC
INTENSITY OF COMPTON SCATTERING   191.44 CTS/MIN
 OMEGA  CHI   TIME   COSINE  COSINE  RADIUS INTENSITY
 -PHI- -PSI-  ·SEC   DELTA   ALPHA          CTS/MIN
  85.0   0.0   5.52  .08715 0.00000 1.00000  5794.30           1
  85.0   7.5   5.69  .08641  .01138  .98867  5612.96           2
  ...etc.
  85.0 180.1   5.60 -.08715 -.00027  .99972  5707.59          25
  90.0 180.1   3.31 0.00000 0.00000 1.00000  5858.84          26
                                      END OF BIAX SCAN NO.   1
```

TABLE 12

LISTING OF REWORK PROGRAM

```
C   C
C           REWORK PROGRAM      FORGO COMPILER      OCTOBER 1964
C           FOR RECALCULATING INDIVIDUAL CARDS FROM BIAXIAL SCAN
  1     READ 80,AB,COS2,BKNBR,SCNBR
        READ 81,FDEG,FMIN,BT,COMPT,THK
        ABTH-AB*THK
        THET=0.5*(FDEG+FMIN/60.)
        THET=3.1415927*(THET/180.)
        COST-ABSF(COSF(THET))
        COS2T=COSF(2.*THET)
        CPOL-(1.+COS2)/(1.+COS2*COS2T*COS2T)
        PUNCH 84,FDEG,FMIN
  23    READ 81,PHID,PSID,TEXP
        IF (TEXT) 1, 1, 110
  110   PHI=3.1415927*(PHID/180.)
        PSI=3.1415927*(PSID/180.)
  19    COSA=SINF(PSI)*COSF(PHI)
        RAD=SQRTF((1.0-ABSF(COSA))/(1.0+ABSF(COSA)))
C       TEST FOR TILT
        TEST=PHID*(180.-ABSF(PHID))*(360.-ABSF(PHID))
        IF (TEST) 15,7,15
C       TESTS FOR REFLECTION OR TRANSMISSION
  15    COSP=COSF(THET+PHI)
        COSM=COSF(THET-PHI)
        TEST=COSP*COSM
        IF (TEST) 16,12,13
  12    PUNCH 63,PHID,PSID,TEXP
        GO TO 23
C       UNTILTED SAMPLE
  7     CABS=COST*EXPF(ABTH/COST)
        TEST=COSF(PSI)*COSF(PHI)
        IF (TEST) 17,4,18
  17    COSD=-1.0
        GO TO 8
  4     COSD=0.0
        GO TO 8
  18    COSD=1.0
        GO TO 8
C       TRANSMISSION METHOD
  13    SECP=1.0/ABSF(COSP)
        SECM=1.0/ABSF(COSM)
        SECD=SECP-SECM
        CABS=(ABTH*SECD)/(SECM*(EXPF(-ABTH*SECM)-EXPF(-ABTH*SECP)))
        GO TO 14
C       REFLECTION METHOD
  16    SECP=1.0/ABSF(COSP)
        SECM=1.0/ABSF(COSM)
        SECS=SECP+SECM
        CABS=(ABTH*SEC)/(SECM*(1.0-EXPF(-ABTH*SECS)))
  14    SINA=SQRTF(1.0-COSA*COSA)
        IF (SINA) 2,4,2
  2     COSD-COSF(PSI)*COSF(PHI)/SINA
C       COMPUTATION OF CORRECTED INTENSITY AND PUNCHING OF RESULTS
  8     DIF=60.*(SCNBR/TEXP-BKNBR/BT)
        CINT=DIF*CABS*CPOL-COMPT
```

TABLE 12 (cont.)

```
        PUNCH 49,PHID,PSID,TEXP,COSD,COSA,RAD,CINT
        GO TO 23
  111   CONTINUE
   49   FORMAT (1X,2F6.1,F8.2,3F8.5,F9.2,13X,I5)
   63   FORMAT (1X,2F6.1,F8.2,34X,23H OMITTED AS IMPOSSIBLE )
   80   FORMAT (7X,2F6.4,6X,2F6.0)
   81   FORMAT (1X,5F6.2)
   84   FORMAT (21H CORRECTED CARDS FOR F6.1,5H DEG F6.1,5H MIN )
        END
```

TABLE 13

EXAMPLE OF INPUT DATA, REWORK PROGRAM

```
0.      11.3  1.00 1.5418 1000. 1000.
21.    20.    398.61191.44.0096
85.     7.5   5.69
```

TABLE 14

EXAMPLE OF OUTPUT DATA, REWORK PROGRAM

```
C  C
 CORRECTED CARDS FOR   21.0 DEG   20.0 MIN
    85.0   7.5    5.69  .08642  .01138  .98869  5612.97
```

TABLE 15.   LISTING OF POLE FIGURE CONTOUR PROGRAM

```
*      JOB           POLE FIGURE CONTOUR PROGRAM   OCTOBER 1964
C                         KINGSTRAN II
       DIMENSION CHI(19,25),TEXP(19,25),CINT(19,25),CONT(15),OMEGA(19)
       DIMENSION TILT(6),X(6),TRIP(12)
  49   FORMAT (1X,2F6.1,F8.2,3F8.5,F9.2,13X,I5)
  53   FORMAT (1X,12F6.2)
  54   FORMAT (72H
      1                                       )
  57   FORMAT (56X,16H END OF MAP NO. I3)
  58   FORMAT (25X,38H POLE FIGURE CONTOUR PROGRAM - MAP NO. I3/)
  59   FORMAT (65H THE FOLLOWING INPUT DATA CARDS HAVE BEEN CORRECTED AS
      1INDICATED /72H  OMEGA  CHI    TIME   COSINE  COSINE  RADIUS INTENSI
      2TY  FORMER      CARD/63H  -PHI- -PSI-   SEC     DELTA   ALPHA
      3    CTS/MIN     VALUE )
  60   FORMAT (29H NO LOCATION FOUND FOR OMEGA=F6.1)
  61   FORMAT (10X/14H LOCATIONS OF F9.2,22H CTS/MIN CONTOUR LINE /13H  O
      1MEGA  CHI )
  62   FORMAT (1X,2F6.1 )
  64   FORMAT (1X,2F6.1,F8.2,3F8.5,2F9.2,5X,I4)
  66   FORMAT (32H INPUT DATA NOT IN PROPER ORDER  )
  67   FORMAT (29H INPUT DATA READ FOR MAP NO. I3)
  68   FORMAT (20H SCAN DECK ACCEPTED  )
C                        SETTING OF CONTOUR LEVELS
       CONT(1)=100.
       CONT(2)=160.
       CONT(3)=250.
       CONT(4)=400.
       CONT(5)=640.
       DO 303 K=6,15
       CONT(K)=10.*CONT(K-5)
 303   CONTINUE
       L=0
C              READIN OF DIMENSIONS OF DATA MATRICES
 300   READ 54
       READ 53,FNOMG,FNCHI,CONTLO,CONTHI
       NOMG=FNOMG
       NCHI=FNCHI
C                  MATRICES FILLED WITH ZEROES
       DO 301 I=1,19
       DO 302 J=1,25
       CHI(I,J)=0.
       TEXP(I,J)=0.
       CINT(I,J)=0.
 302   CONTINUE
 301   CONTINUE
       SWITCH=0.
C                  SETTING OF OMEGA LEVELS
       READ 53,(TRIP(NBR),NBR=1,12)
       OMEGA(1)=0.
       DO 710 I=2,NOMG
       OMEGA(I)=OMEGA(I-1)+5.
 713   DO 711 NBR=1,12
       TEST=OMEGA(I)-TRIP(NBR)
       IF (TEST) 711,712,711
 712   OMEGA(I)=OMEGA(I)+5.
       GO TO 713
 711   CONTINUE
```

```
 710    CONTINUE
C                        READIN OF CORRECTING FACTORS FOR CERTAIN OMEGA VALUES
C                        IF NO SUCH CORRECTING IS DESIRED READ A BLANK CARD
        READ 53,(TILT(M),X(M),M=1,6)
        L=L+1
        PUNCH 58,L
        TYPE 58,L
        PUNCH 54
        TYPE 54
        DO 305 M=1,6
        IF (X(M)) 305,306,305
  306   NCORXN=M-1
        GO TO 350
  305   CONTINUE
        NCORXN=6
                        READIN OF DATA DECK - OBTAINED FROM BIAXIAL SCAN PGM
  350   DO 704 I1=1,NOMG
        DO 702 J=1,NCHI
        READ 49,PHI,PSI,TIME,COSD,COSA,RAD,RATE,KARD
        IF (NCORXN) 310,310,311
                        CORRECTING OF SELECTED INTENSITY VALUES
  311   DO 314 M=1,NCORXN
        TEST=PHI-TILT(M)
        IF (TEST) 314,316,314
  316   IF (SWITCH) 315,317,315
C                        PUNCHOUT OF CORRECTED INPUT DATA CARDS
  317   PUNCH 59
        SWITCH=1.
  315   OLDINT=RATE
        RATE=RATE*X(M)
        PUNCH 64,PHI,PSI,TIME,COSD,COSA,RAD,RATE,OLDINT,KARD
  314   CONTINUE
  310   CONTINUE
C                        ASSIGNING LOCATIONS ACCORDING TO OMEGA VALUE
        DO 335 I=1,NOMG
        TEST=OMEGA(I)-ABSF(PHI)
        IF (TEST) 701,700,701
  701   TEST=OMEGA(I)-ABSF(180.-PHI)
        IF (TEST) 335,700,335
  335   CONTINUE
        TYPE 60,PHI
        STOP
  700   CHI(I,J)=PSI
        TEXP(I,J)=TIME
        CINT(I,J)=RATE
C                        DATA POINT AT FILM NORMAL ORIENTATION IS DUPLICATED
C                        ACROSS THE MATRICES
        TEST=(90.-PHI)*(270.-PHI)
        IF (TEST) 702,703,702
  703   DO 705 J1=J,NCHI
        CHI(I,J1)=PSI
        TEXP(I,J1)=TIME
        CINT(I,J1)=RATE
  705   CONTINUE
        GO TO 704
  702   CONTINUE
  704   CONTINUE
        TYPE 68,L
C                        CHI VALUES CHANGED TO PROPER RANGE
        DO 318 I=1,NOMG
        DO 319 J=1,NCHI
```

```
321     IF (CHI(I,J)) 380,319,381
380     CHI(I,J)=CHI(I,J)+180.
        GO TO 321
381     TEST=CHI(I,J)-180.
        IF (TEST) 319,382,382
382     CHI(I,J)=TEST
        GO TO 381
319     CONTINUE
318     CONTINUE
        DO 4 I=1,NOMG
322     TEST=10.-CHI(I,NCHI)
        IF (TEST) 4,5,5
5       CHI(I,NCHI)=CHI(I,NCHI)+180.
4       CONTINUE
C                       CHI VALUES AT OMEGA=90 ARE ARBITRARILY ASSIGNED THE
C                       SAME VALUES THAT APPEAR IN ADJACENT ROW OF CHI MATRIX
        TEST=90.-OMEGA(NOMG)
        IF (TEST) 550,551,550
551     DO 552 J=1,NCHI
        CHI(NOMG,J)=CHI(NOMG-1,J)
552     CONTINUE
550     CONTINUE
C                       VERIFICATION THAT CHI VALUES INCREASE OR DECREASE
C                       MONOTONICALLY AT FIXED OMEGA VALUE
        DO 339 I=1,NOMG
        DO 340 J=3,NCHI
        TEST=(CHI(I,J)-CHI(I,J-1))*(CHI(I,J-1)-CHI(I,J-2))
        IF (TEST) 341,341,340
341     TYPE 66
        STOP
340     CONTINUE
339     CONTINUE
C                       CHI MATRIX CHECKED TO SEE THAT CHI VALUES ARE NEARLY
C                       EQUAL FOR ADJACENT ELEMENTS OF A COLUMN
        DO 730 I=2,NOMG
        DO 731 J=1,NCHI
        TEST=5.-ABSF(CHI(I,J)-CHI(I-1,J))
        IF (TEST) 732,731,731
731     CONTINUE
730     CONTINUE
        GO TO 733
732     TYPE 66
        STOP
733     TYPE 88
        SWITCH=0.
C                       START OF MAIN CONTOUR MAPPING LOOP
        DO 6 K=1,15
        TEST=CONT(K)-CONTLO
        IF (TEST) 6,630,630
630     IF (CONTHI) 631,631,632
632     TEST=CONT(K)-CONTHI
        IF (TEST) 631,631,6
631     CONTINUE
C                       START OF FIRST INTERPOLATION SUBLOOP
        DO 16 I=1,NOMG
        JZERO=1
C                       DATA POINT WHERE TEXP IS ZERO IS TO BE AVOIDED
19      IF (TEXP(I,JZERO)) 18,18,17
18      JZERO=JZERO+1
        GO TO 19
```

```
  17     JZERO=JZERO+I
         DO 20 J=JZERO,NCHI
         DELTAI=CONT(K)-CINT(I,J-I)
         IF (TEXP(I,J)) 21,21,22
  22     DELTA2=CONT(K)-CINT(I,J)
         TEST=DELTAI*DELTA2
         IF (TEST) 100,100,20
  100    IF (DELTAI) 23,24,23
  24     OMGX=OMEGA(I)
         CHIX=CHI(I,J-I)
  28     IF (SWITCH) 324,323,324
  323    PUNCH 61,CONT(K)
         SWITCH=I.
C                      PUNCHOUT OF CONTOUR LOCATION
  324    PUNCH 62,OMGX,CHIX
         GO TO 20
  23     IF (DELTA2) 25,26,25
  26     OMGX=OMEGA(I)
         CHIX=CHI(I,J)
         GO TO 28
  25     FRACTN=DELTAI/(DELTAI-DELTA2)
  390    SPAN=ABSF(CHI(I,J)-CHI(I,J-I))
C                      IF CHI SPAN IS TOO GREAT NO INTERPOLATION IS MADE
         TEST=16.-SPAN
         IF (TEST) 20,20,391
  391    CONTINUE
         DELCHI=FRACTN*(CHI(I,J)-CHI(I,J-I))
         CHIX=CHI(I,J-I)+DELCHI
         OMGX=OMEGA(I)
         GO TO 28
  21     TEST=NCHI-J
         IF (TEST) 20,20,325
  325    J=J+I
         IF (TEXP(I,J))21,21,20
  20     CONTINUE
  16     CONTINUE
C                      START OF SECOND INTERPOLATION SUBLOOP
         DO 36 J=1,NCHI
         IZERO=1
C                      DATA POINT WHERE TEXP IS ZERO IS TO BE AVOIDED
  39     IF (TEXP(IZERO,J)) 38,38,37
  38     IZERO=IZERO+I
         GO TO 39
  37     IZERO=IZERO+I
         DO 40 I=IZERO,NOMG
         DELTAI=CONT(K)-CINT(I-I,J)
         IF (TEXP(I,J)) 41,41,42
  42     DELTA2=CONT(K)-CINT(I,J)
         TEST=DELTAI*DELTA2
         IF (TEST) 200,40,40
  200    FRACTN=DELTAI/(DELTAI-DELTA2)
         DELOMG=FRACTN*(OMEGA(I)-OMEGA(I-I))
         DELCHI=FRACTN*(CHI(I,J)-CHI(I-I,J))
         CHIX=CHI(I-I,J)+DELCHI
         OMGX=OMEGA(I-I)+DELOMG
  48     IF (SWITCH) 328,327,328
  327    PUNCH 61,CONT(K)
         SWITCH=I.
C                      PUNCHOUT OF CONTOUR LOCATION
  328    PUNCH 62,OMGX,CHIX
         GO TO 40
```

```
41      TEST=NOMG-I
        IF (TEST) 40,40,326
326     I=I+1
        IF (TEXP(I,J)) 41,41,40
40      CONTINUE
36      CONTINUE
        SWITCH=0.
6       CONTINUE
15      TYPE   57,L
        PUNCH 57,L
        GO TO 300
        END
X       EOJ
```

TABLE 16

EXAMPLE OF INPUT DATA, POLE FIGURE CONTOUR PROGRAM

```
        SAMPLE 32G  110 PLANE DATA          FOR TESTING CONTOUR PROGRAM
17.   25.   500.  20000.
75.   80.
-70.  1.
   90.0 180.1    6.78 0.00000 0.00000 1.00000   2865.86
   85.0   0.0   30.46  .08715 0.00000 1.00000    885.83
   85.0   7.5   29.04  .08642  .01138  .98869    942.20
   85.0  15.0   27.11  .08420  .02257  .97766   1028.27
   85.0  22.5   27.93  .08055  .03338  .96715    990.25
```

TABLE 17

EXAMPLE OF OUTPUT, POLE FIGURE CONTOUR PROGRAM

```
                    POLE FIGURE CONTOUR PROGRAM    MAP NO.  1          0002
                                                                       0003
        SAMPLE 32G  110 PLANE DATA          FOR TESTING CONTOUR PROGRAM  0004
THE FOLLOWING INPUT DATA CARDS HAVE BEEN CORRECTED AS INDICATED        0005
OMEGA  CHI    TIME   COSINE  COSINE  RADIUS INTENSITY FORMER           0006
-PHI- -PSI-   SEC    DELTA   ALPHA          CTS/MIN  VALUE       CARD   0007
-70.0   .0    6.47  .34202  .00000 1.00000  2349.53  2349.53      0    0008
-70.0  7.5    5.83  .33942  .04468  .95626  2631.67  2631.67      0    0009
...etc.
-70.0 180.1   6.92 0.34201 0.00107  .99892  2182.40  2182.40      0    0032
                                                                       0033
LOCATIONS OF   640.00 CTS/MIN CONTOUR LINE                            0034
OMEGA  CHI                                                            0035
  85.0  44.9                                                          0036
  85.0 119.6                                                          0037
  ...etc.                                                             0038

LOCATIONS OF  1000.00 CTS/MIN CONTOUR LINE                            0059
OMEGA  CHI                                                            0060
  85.0  12.5                                                          0061
  ...etc.                                          END OF MAP NO. 1
```

TABLE 18.

SUBROUTINES AND FUNCTIONS USED IN RADIAL AND BIAXIAL SCAN PROGRAMS

```
        SUBROUTINE INTER
900     FORMAT(34HINDEPENDENT VARIABLE OUTSIDE RANGE)
        DIMENSION Y(7.6),ANS(6),X(7)
        COMMON Y,F,ANS,HYD,CAR,XNI,OXY,FLO,CHL
        DO 5 I=1,7
        X(I)=I-I
    5   X(I)=X(I)/10.0
        IF(F)1,2,31
    1   TYPE 900
        STOP
    2   J=1
    3   DO 6 I=1,6
    6   ANS(I)=Y(J,I)
        GO TO 50
   31   IF(F-0.1)7,8,19
    8   J=2
        GO TO 3
    7   K=1
        GO TO 60
   19   DO 12 I=3,7
   11   IF(F-X(I))9,13,12
   13   J=I
        GO TO 3
    9   K=I-2
        GO TO 60
   60   N=K+3
        DO 40 L=1,6
        YY=0.0
        DO 31 I=K,N
        PRO = 1.0
        DO 20 J=K,N
        IF(I=J)10,20,10
   10   PRO =PRO*(F -X(J))/(X(I)=X(J))
   20   CONTINUE
        YY=YY+PRO*Y(I,L)
   30   CONTINUE
   40   ANS(L)=YY
   50   HYD=ANS(1)
        CAR=ANS(2)
        XNI=ANS(3)
        OXY=ANS(4)
        FLO=ANS(5)
        CHL=ANS(6)
        RETURN

        FUNCTION RADIAN(THETD)
        RETURN

        FUNCTION DECML(A,B,C,D)
        DECML=4.*A+2.*B+2.*C+D
        RETURN
        END
```

TABLE 19

NAMES OF THE VARIOUS RELAYS IN THE CONTROL UNIT

Relay	Name
RE1	Count-Advance Power Relay
RE2	Start Count Relay
RE3	Scaler Stopper
RE4	Advance Holding Relay
RE5	Restorer Slave
RE6	Motor Selector Slave
RE7	Release Disconnect Relay
RE8	Punch Disconnect Relay
RE9	Stepper Verify Relay
RE10	Stepper Starter
RE11	Stepper Stopper
RE12	Relaxation Oscillator
RE13	Card Releaser
RE14	Digit Selector
RE15	Punch Start Relay
RE16	Keyboard Relay
RE17	End of Count Relay
RE18	Electronic Relay
RE19	Register Driver
RE20	Motor Selector Slave
RE21	Start Motor Relay
RE22	Stop Motor Relay
RE23	Homing Relay
RE24	Motor Selector Relay
RE25	Timer Hold Relay
RE26	Astable Multivibrator Relay
RE27	Relaxation Oscillator Slave
SR1	Punch Stepping Relay
SR2	Advance Stepping Relay

TABLE 20

LAYOUT OF PUNCH CONTROL UNIT

As seen from above:

RE1	RE4	RE11	12AU7	
RE2	RE5	RE12	RE14	SR1
RE3	RE6-RE10	RE13	RE15	

Two relays, RE25 and RE27, are mounted out of sight against the side of the chassis.

TABLE 21

JACKS ON PUNCH CONTROL UNIT

To Berkeley Scaler:

A. Ground

B. 30V Count

C. 30V End of Count

D. Reset

To Offner Power Supply:

1. +250 VDC

2. +6.3 VDC

3. +500 VDC

4. Ground

5.
   6.3 VAC
6.

To IBM 026 Card Punch:

A. "1" punch line

B. "-" punch line

C. "0" punch line

D. Ground

E. Card release line

F. Restorer coil

G. "+" punch line

H. Restorer coil

I. RE16 coil

J. Keyboard common line

To Advance Control Unit:

1.	2. +250 VDC	3. +30VDC
6.3 VAC		
4.	5. 250V Count	6. 30V Count
7. +500 VDC	8. 250V Advance	9. 30V Advance
10.Digit selector test input	11.250V End of Advance	12.30V Motor Selector
13.Ground	14.250V Punch Complete	15.30V Punch Complete
16.-105 VDC	17.	18.Digit Selector Test Output

TABLE 22

JACKS ON ASTABLE MULTIVIBRATOR

Power Top (octal socket):

1. +200 (unregulated)	5.
2. +375	6.
	6.3 VAC
3.	7.
4. Ground	8. 60VAC between here and pin 1

The remaining jacks all go to the Advance Control Unit

Four-pin Jack:

A. -105 VDC

B.

C. Pulses out

D. Ground

Four-pin Jack:

1.
   Normally open pair
2.

3.
   Normally closed pair
4.

Twelve-pin Jack:

1. Wiggler #1	7. Wiggler #3
2. Normally Closed #1	8. Normally Closed #3
3. Normally Open #1	9. Normally Open #3
4. Wiggler #2	10.
5. Normally Closed #2	11.
6. Normally Open #2	12.

FIGURE 1

GENERAL LAYOUT
OF DIFFRACTOMETER

PLAN VIEW

COPPER TUBES FOR H_2O

NORELCO TUBE TOWER

X-RAY TUBE

SLIT SYSTEM

H.V. CABLE

H.V. CONNECTION (SHEET METAL SHIELDS NOT SHOWN)

BRIDGEPORT ROTARY TABLE

CLAMPS

100°

100°

BEAM STOP (COPPER COVERED WITH LEAD)

SLIT SYSTEM

JEWELER'S LATHE BED

BAIRD-ATOMIC PREAMPLIFIER

GEIGER-MUELLER TUBE
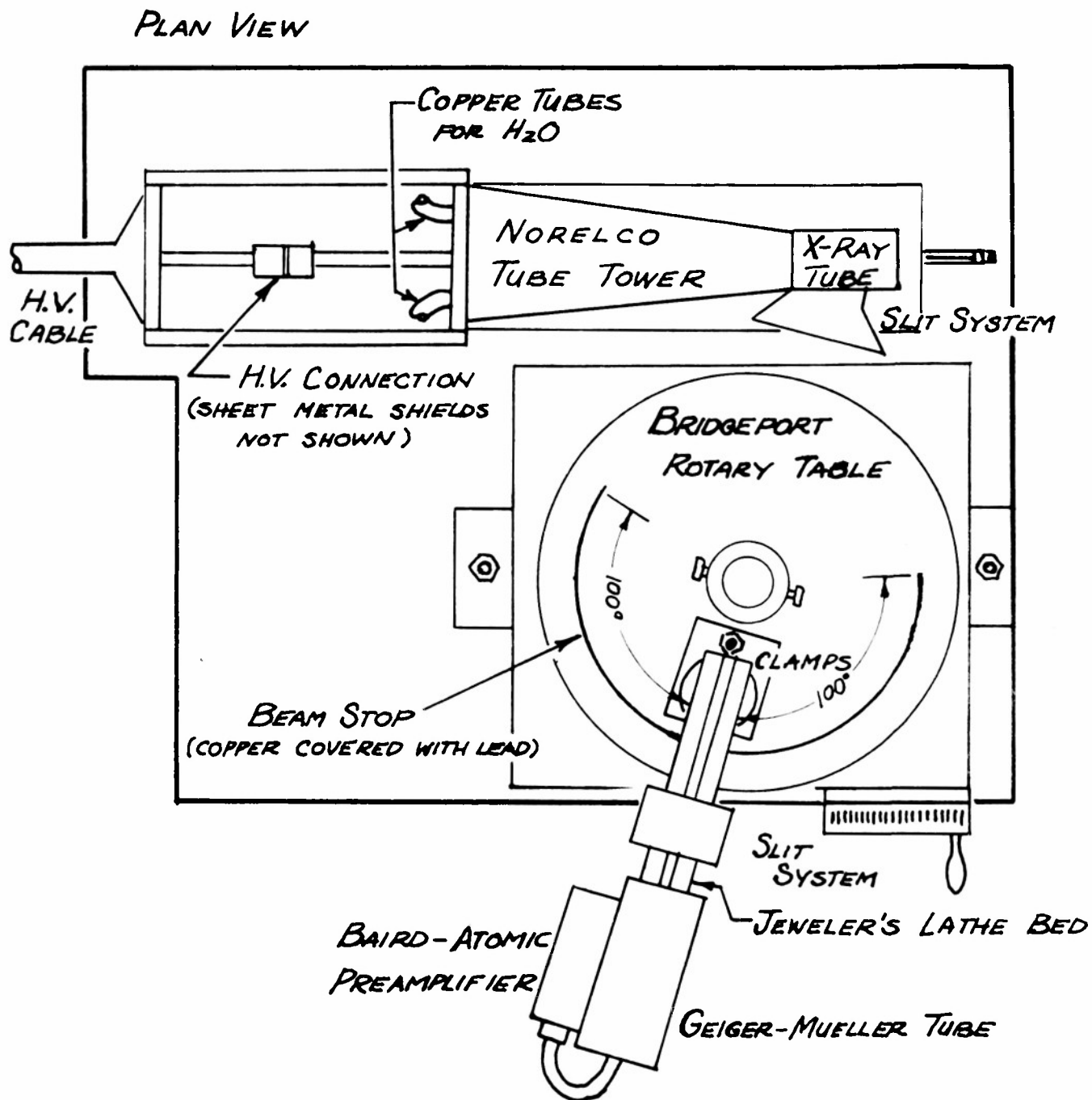
DETAIL OF TUBE STAND
(TUBE TOWER AND CABLES OMITTED)
NOT TO SCALE

.055" SLOTS

BASE

$\frac{1}{4}$-13NC
ALLEN CAP SCREWS

TUBE TOWER

UPRIGHT

$\frac{3}{8}$" SQUARE
STOCK

WINGS

$L\frac{3}{8}"x\frac{9}{16}"$ GROOVE
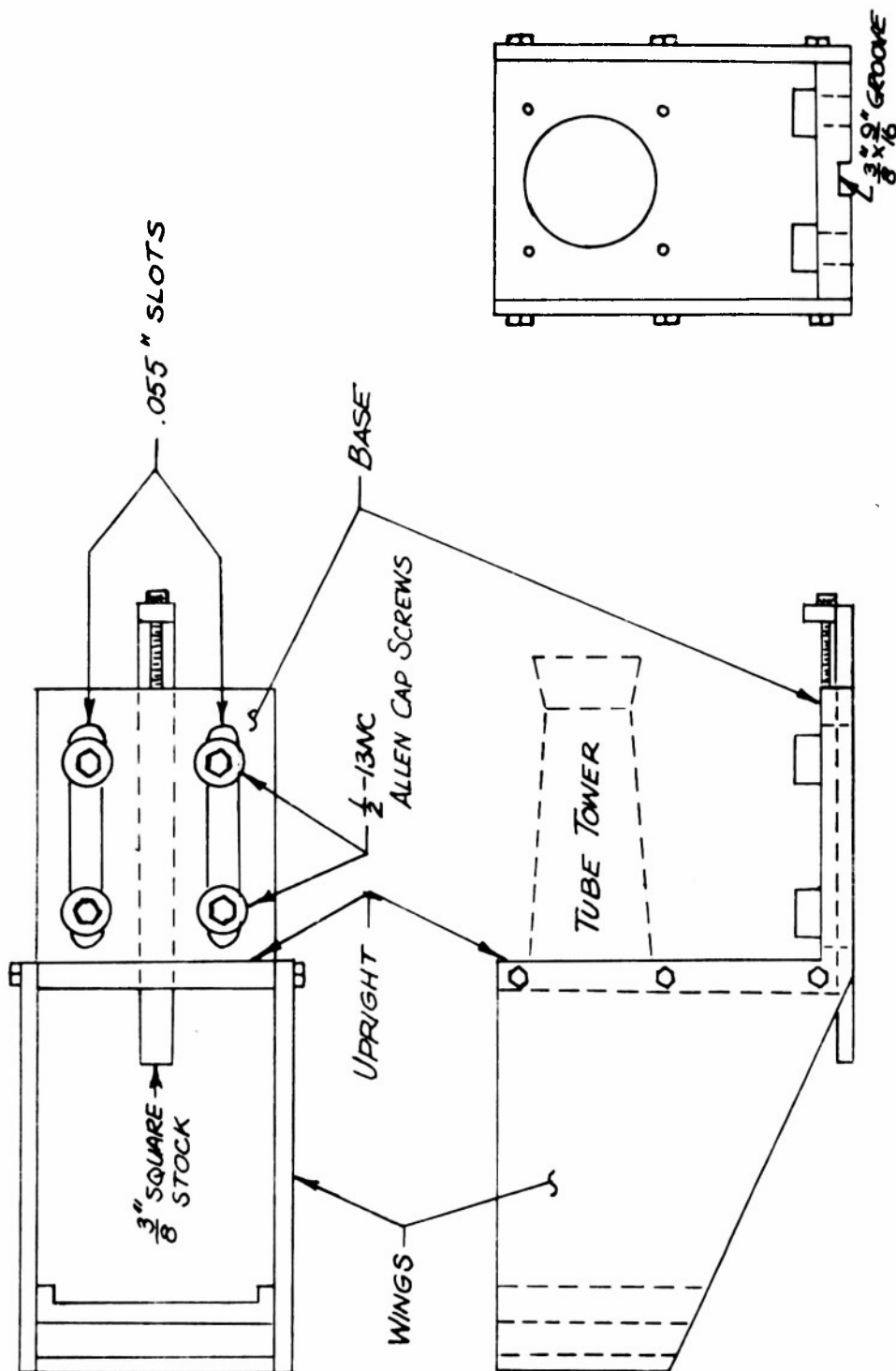
NOTE: SPACE BETWEEN WINGS COVERED WITH
SHEET ALUMINUM (NOT SHOWN HERE) AS
A SAFETY MEASURE.

MATERIAL: ALUMINUM, EXCEPT UPRIGHT AND BASE
WHICH ARE STEEL.

FIGURE 2

SLIT SYSTEM DESIGN

X-RAY LINE SOURCE

DIVERGENCE SOLLER SLIT

DIVERGENCE SLIT

SCATTER SLIT

11 inches

CENTER OF ROTARY TABLE

11 inches

2θ

SCATTER SLIT

RECEIVING SOLLER SLIT

NICKEL FILTER

RECEIVING SLIT

GEIGER-MUELLER TUBE
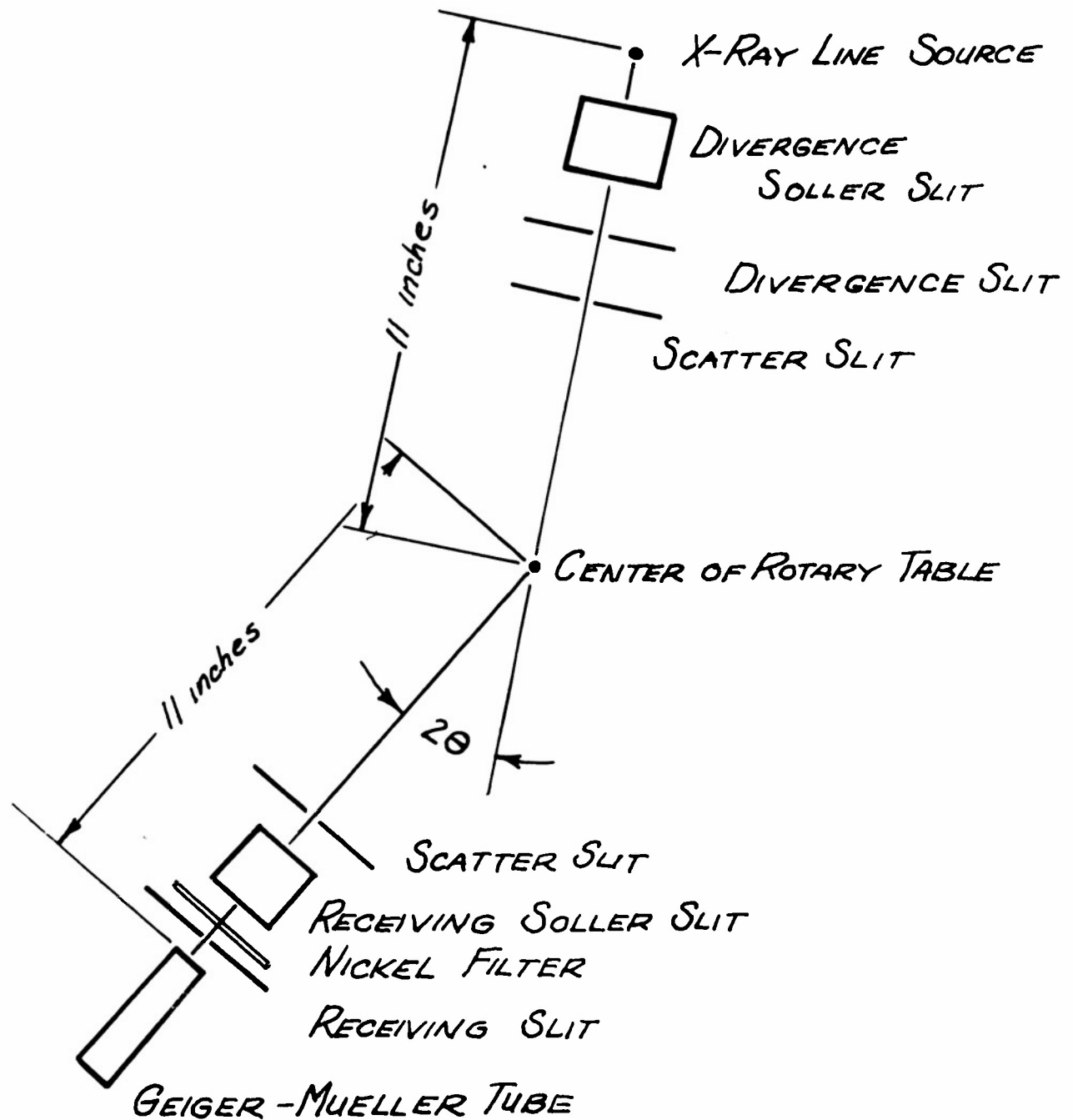
FIGURE 3

Caption for Figures 4a and 4b

TWO VIEWS OF POLE FIGURE GONIOMETER

1. Azimuthal Angle Pulse Motor

2. Tilt Angle Pulse Motor

3. Microswitch

4. Striker Bar

5. Sample
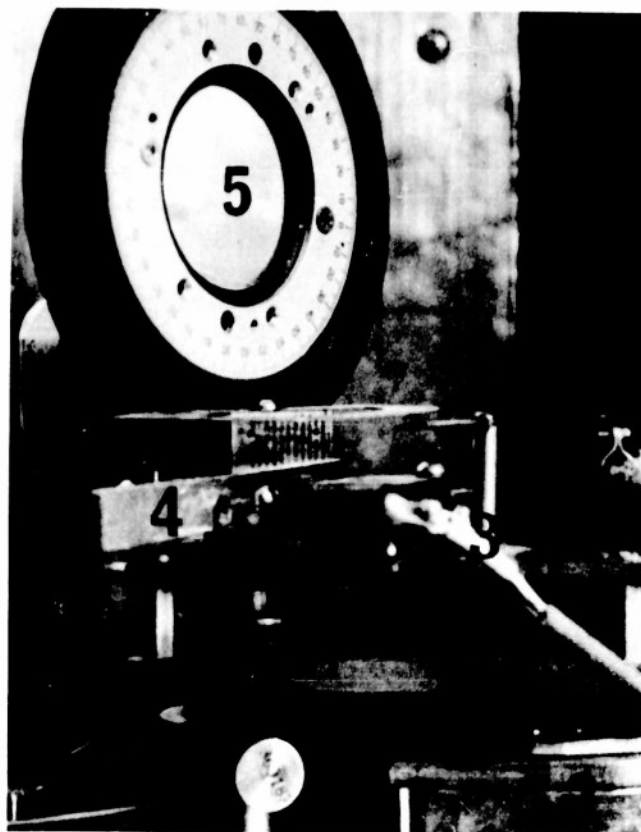
Figure 4a
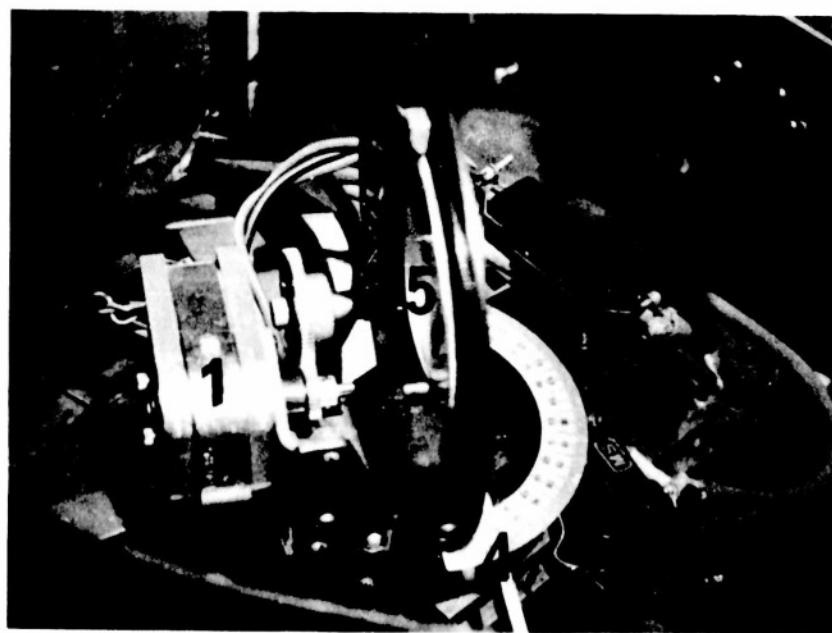
Figure 4b
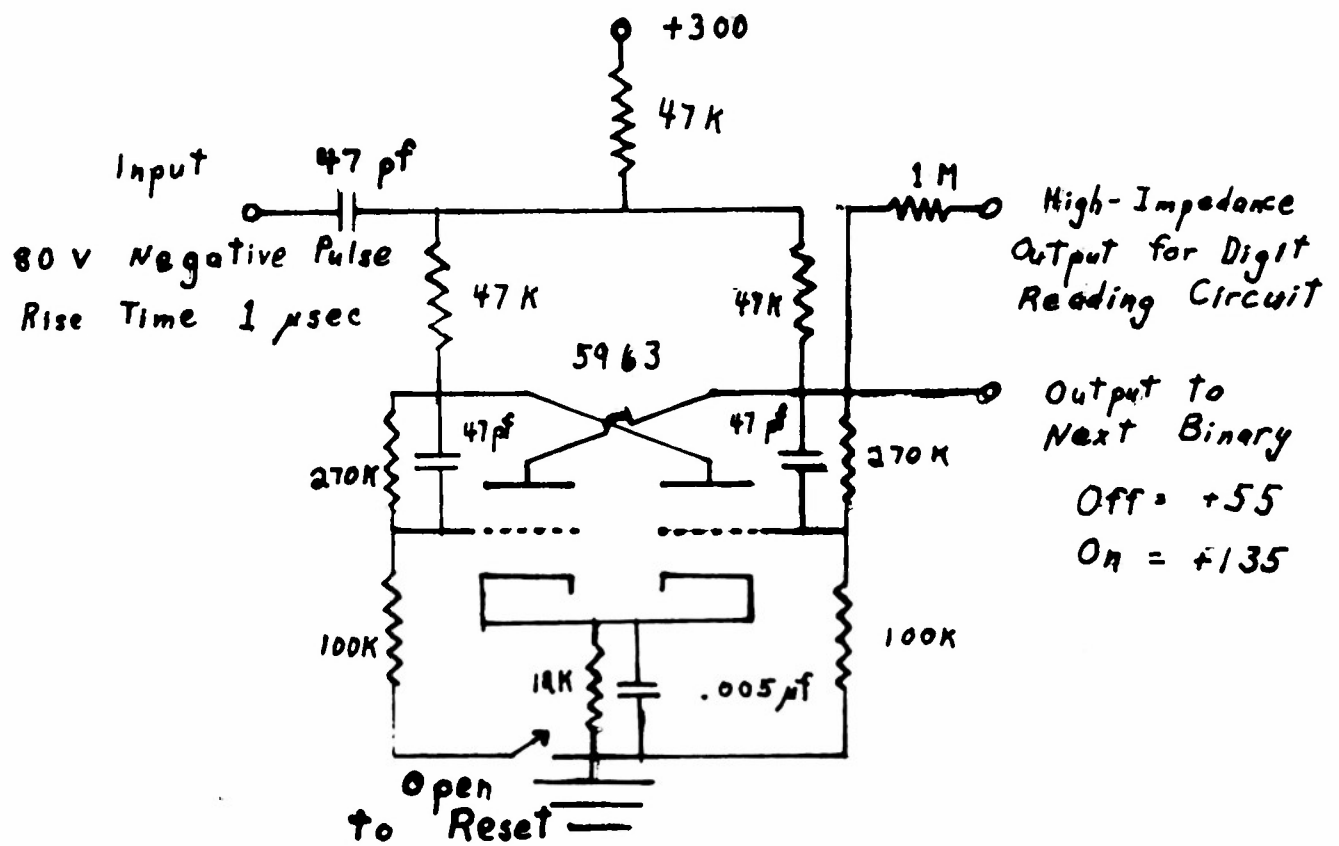
Typical Binary Circuit

Figure 5

ALTERATIONS OF BERKELEY SCALER
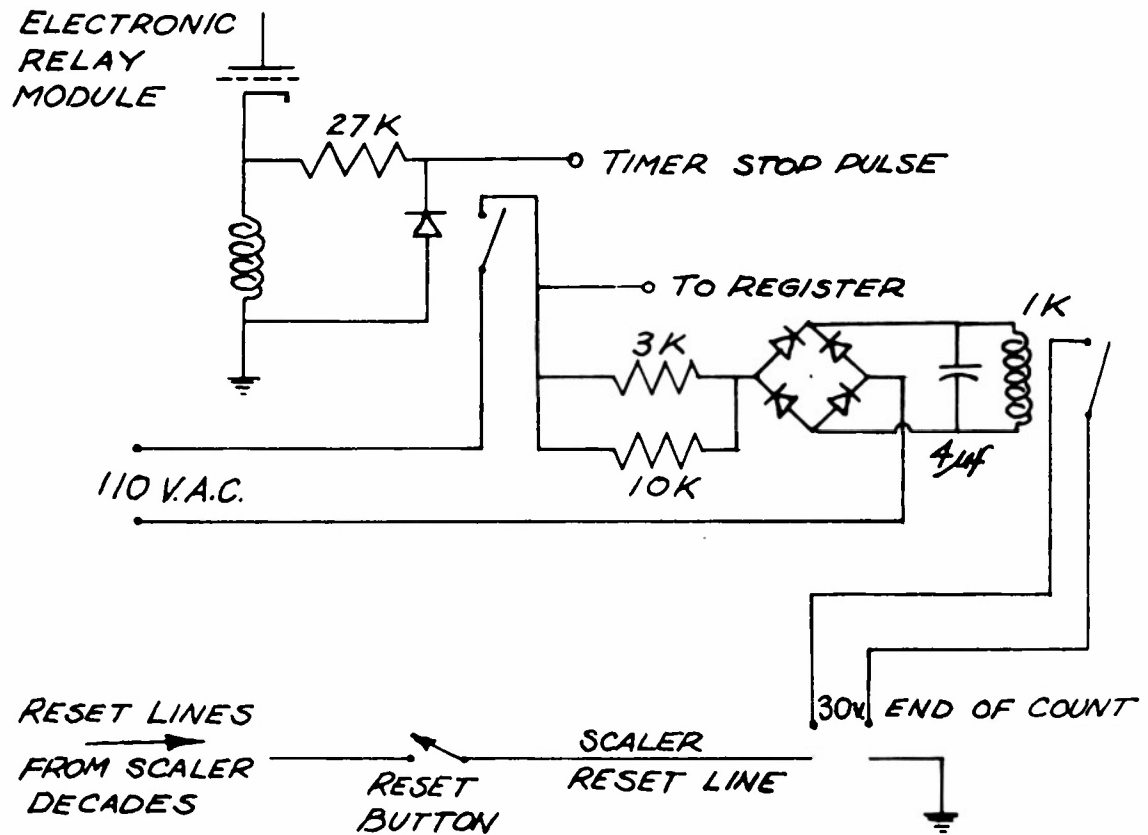
FIGURE 6

RELAY POWER SUPPLY

110 V.A.C.

P.L.

5A.

18
15
9
0
9
15
18

2000 mfd 60 WV

2000 mfd 60 WV

2000 mfd 60 WV

30 V.D.C.
POLARIZED
RECEPTACLE
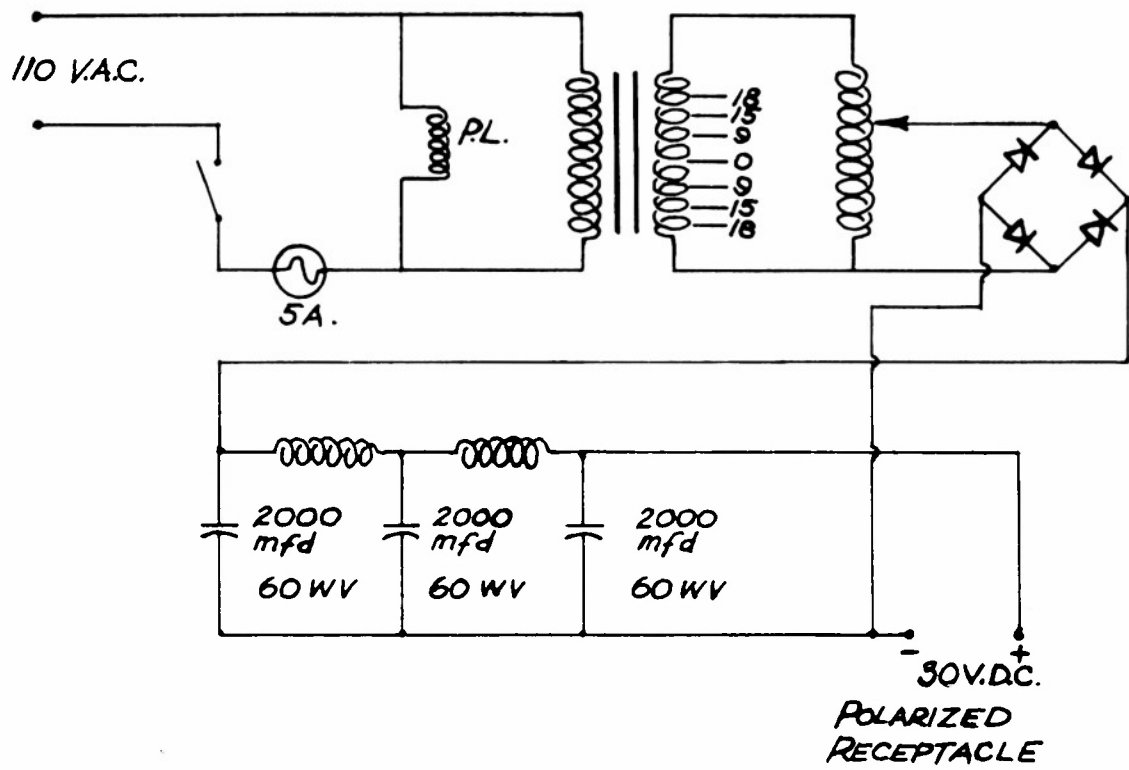
FIGURE 7

PUNCH CONTROL UNIT
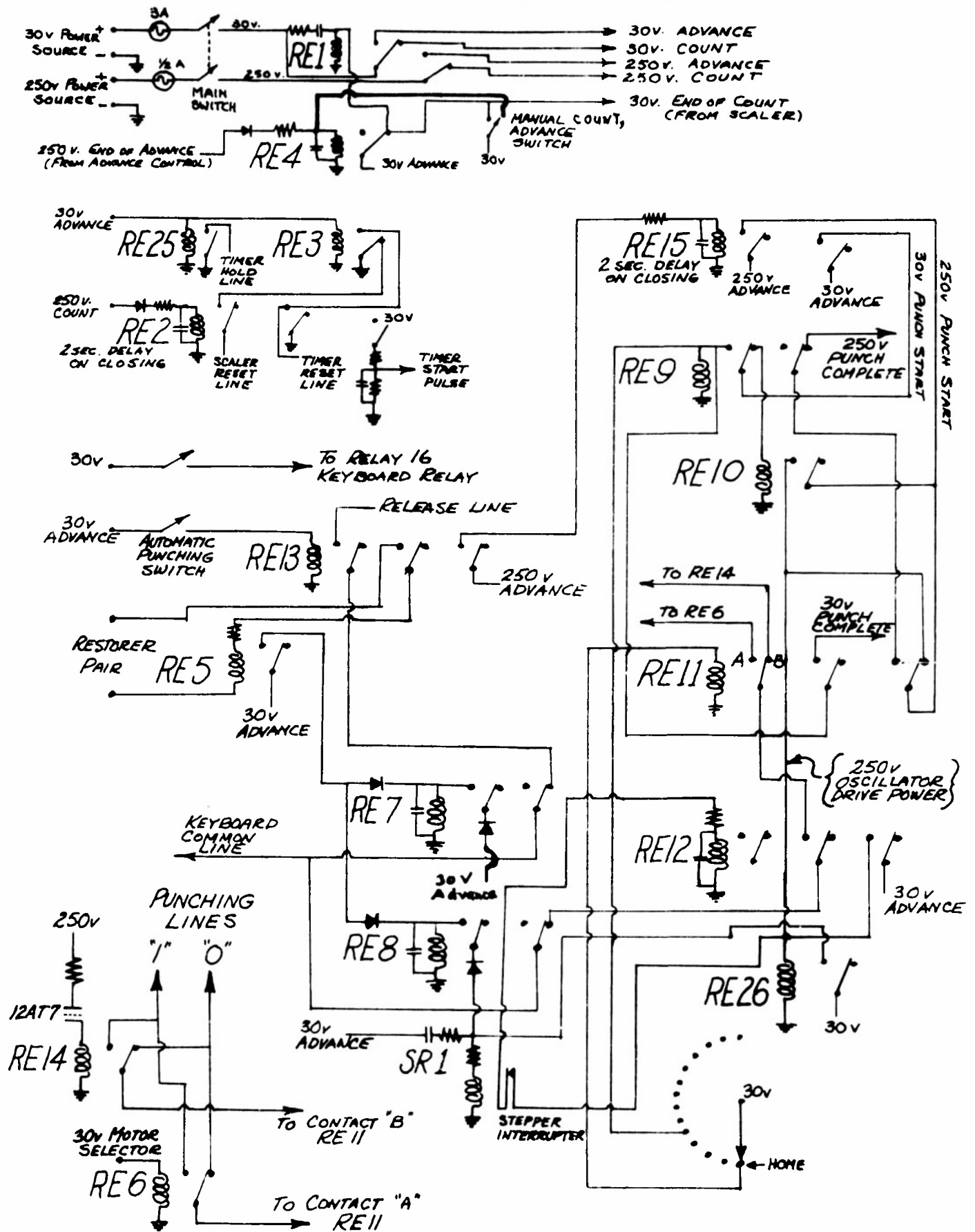
FIGURE 8

ALTERATIONS OF IBM 026 CARD PUNCH

KEYBOARD RELAY

CABLE

RE16

RESTORER

RELEASE

+

INTERRUPTER

−

1

KEYBOARD COMMON

AUTO FEED SWITCH

0

——————— ADDED WIRE

— — — — EXISTING WIRE

- - - - - REMOVED WIRE
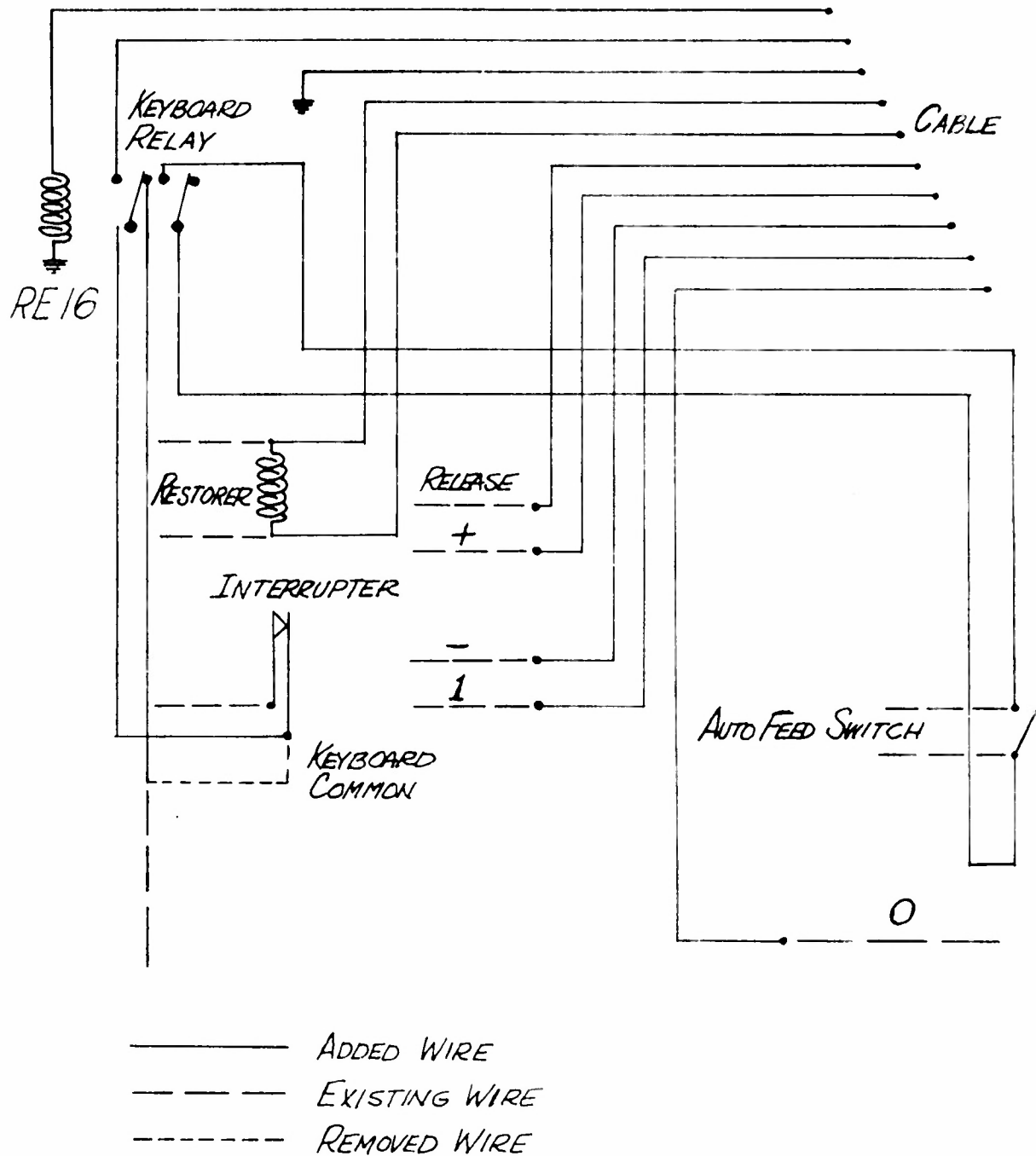
FIGURE 9

Digit   Selector   Circuit

Figure 10

Rotation Dir'n

Bank 7

50

w

w

Cut Here

No Connxn

26

Bank 8

25

Bank 5

50

w

w

26

Bank 6

25

30V Power

To RE-11

To RE-8

S R - 1

Note That Only Half the Wipers Are On A Contact At Any Time.
w = wiper

Bank 1

Bank 3

Second Input Here

Timer wires (at) on This Bank

50

w

w

50

w

26

Bank 4

25

Cut Here →

To Digit Selector

Test Input for Digit Selector

Bank 2

Bank 2 Selector

Adjacent Contacts on Banks 1 & 2 are Jumpered. Do Not Cut These Jumpers.

Provision For Second Input To Reader

Figure 11

ASTABLE MULTIVIBRATOR

FIGURE 12

RE24

MOTOR SELECTOR
RELAY

30V START MOTOR

JUMPER

TILT MOTOR
POWER

AZ. MOTOR
POWER

RE26

ASTABLE MULTIVIBRATOR
RELAY

30V

SPARK
SUPPRESSORS

30V

30V

TILT MOTOR

AZIMUTHAL MOTOR
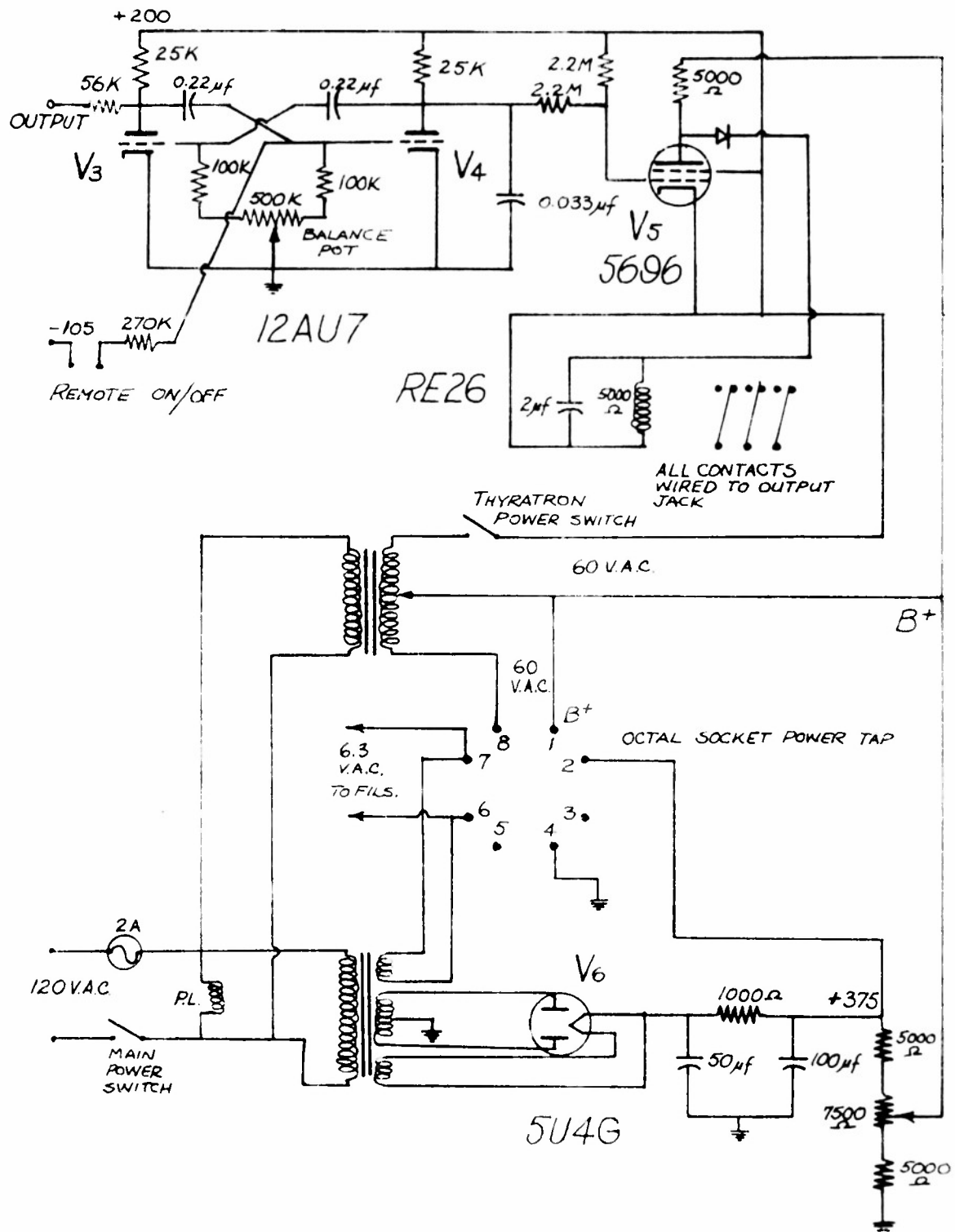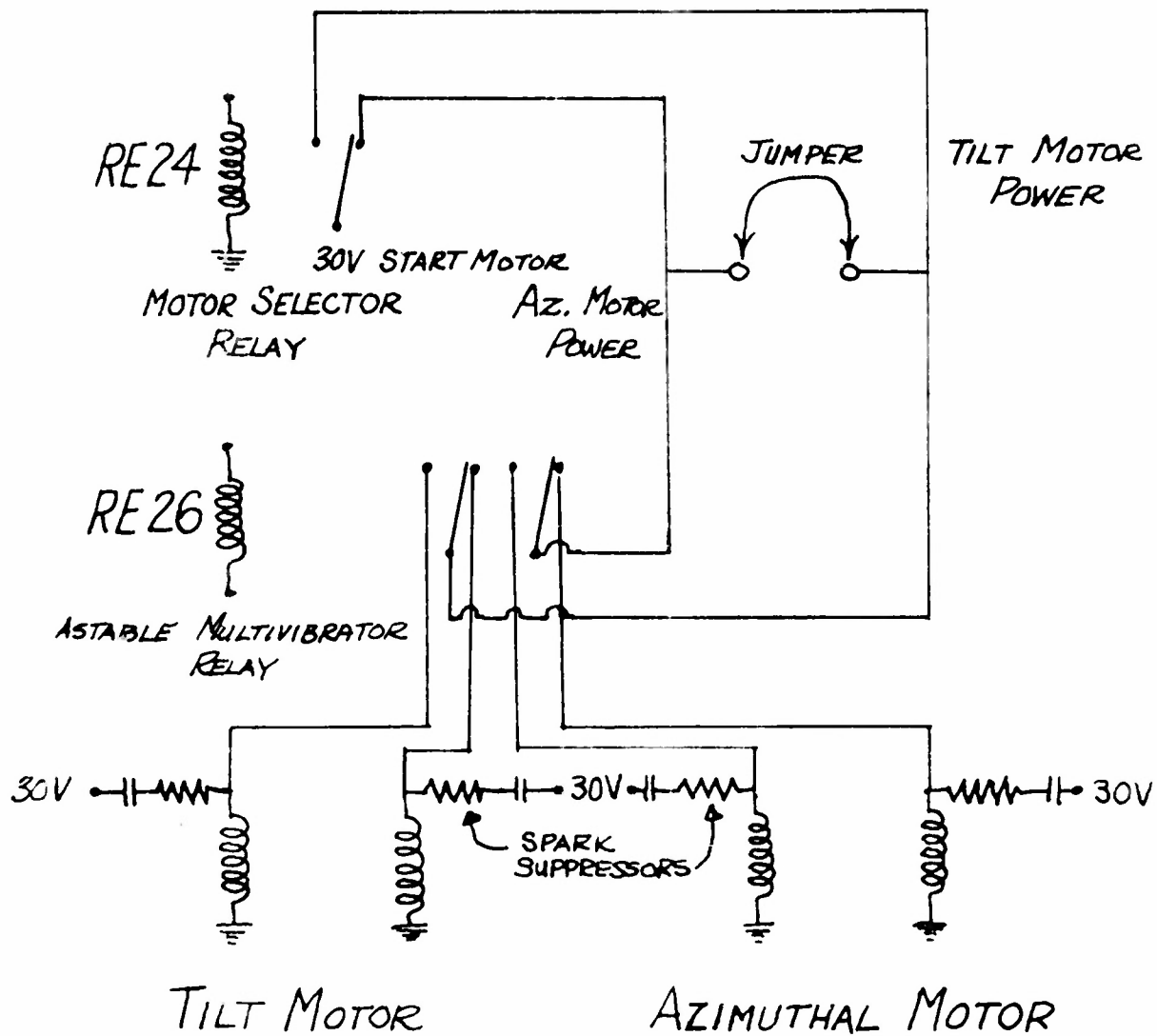
WIRING OF PULSE MOTORS

FIGURE 13
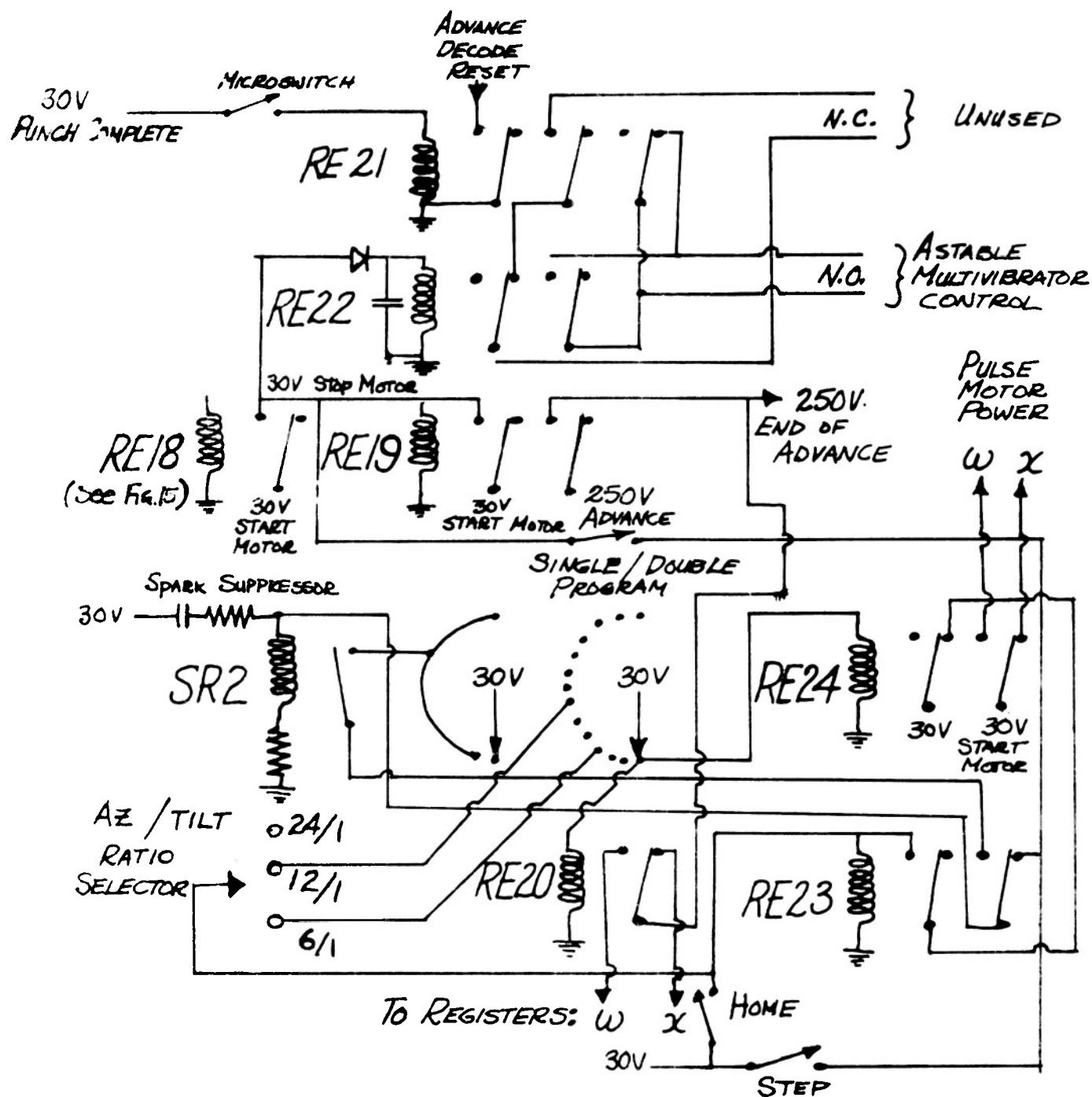
ADVANCE CONTROL UNIT

FIGURE 14

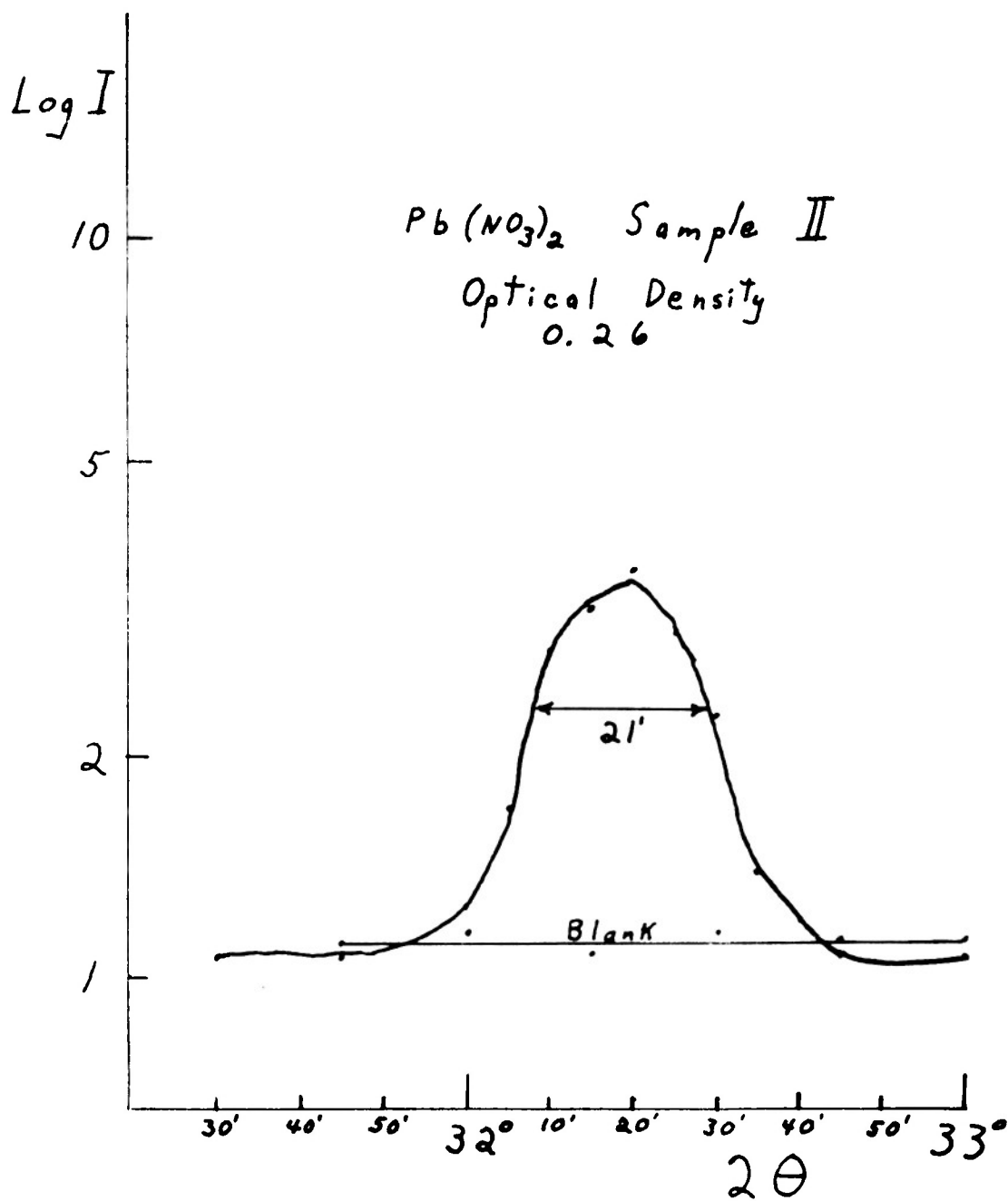ADVANCE DECADE MODULE

ADVANCE PULSE COUNTING CIRCUITS

FIGURE 15

ZEROING SCANS

FIGURE 16
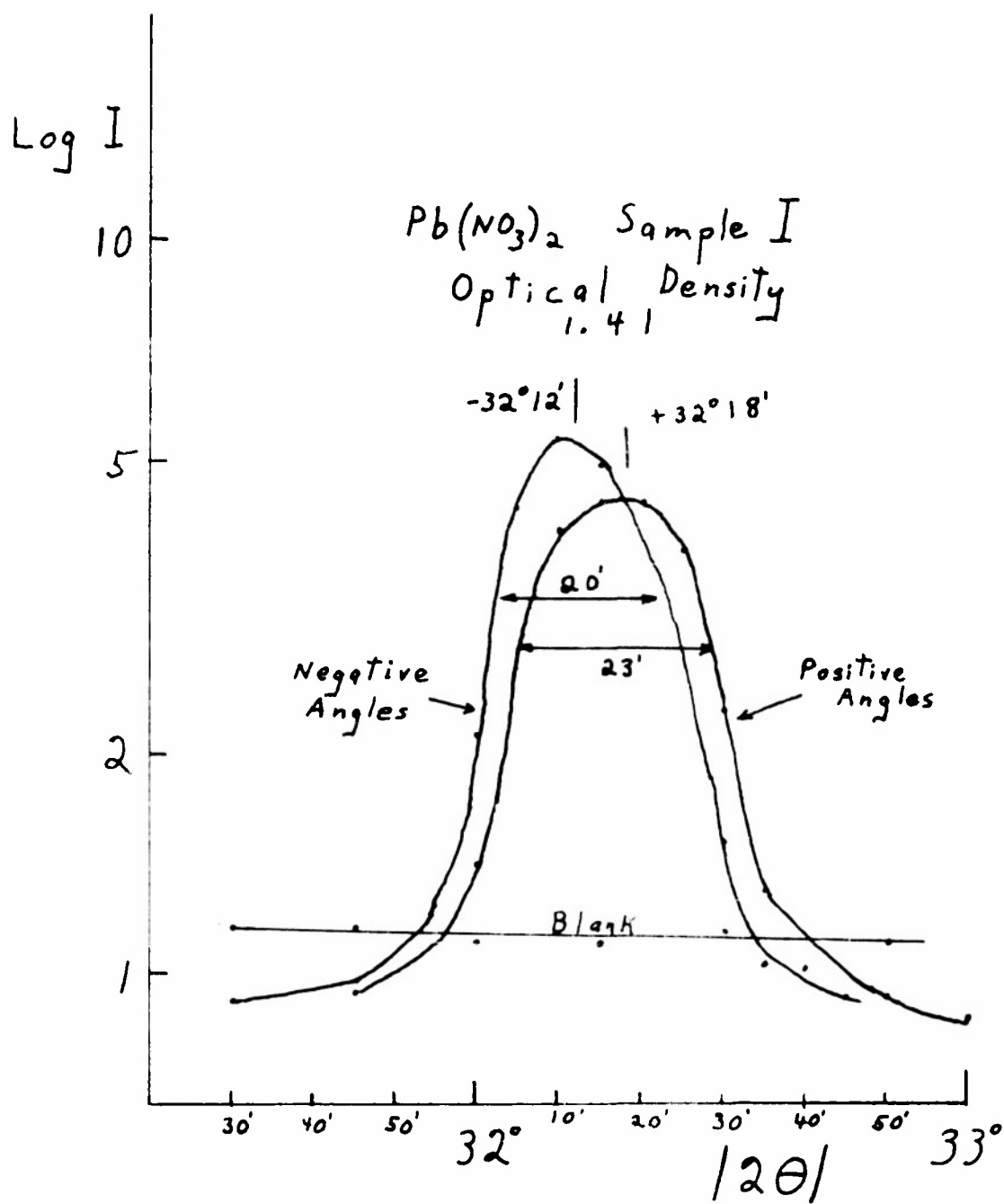
Pb(NO₃)₂ Sample II
Optical Density 0.26

INSTRUMENTAL BROADENING

FIGURE 17

Three Radial Scans - Polyethylene

Sample	Symbol	Thickness
1	.	.0948"
2	o	.0626"
3	x	.0363"

Reflections Indexed by Bunn[5]

$I \times 10^3$
cts/min

110

200

210

020

110

310

Figure 18

2θ
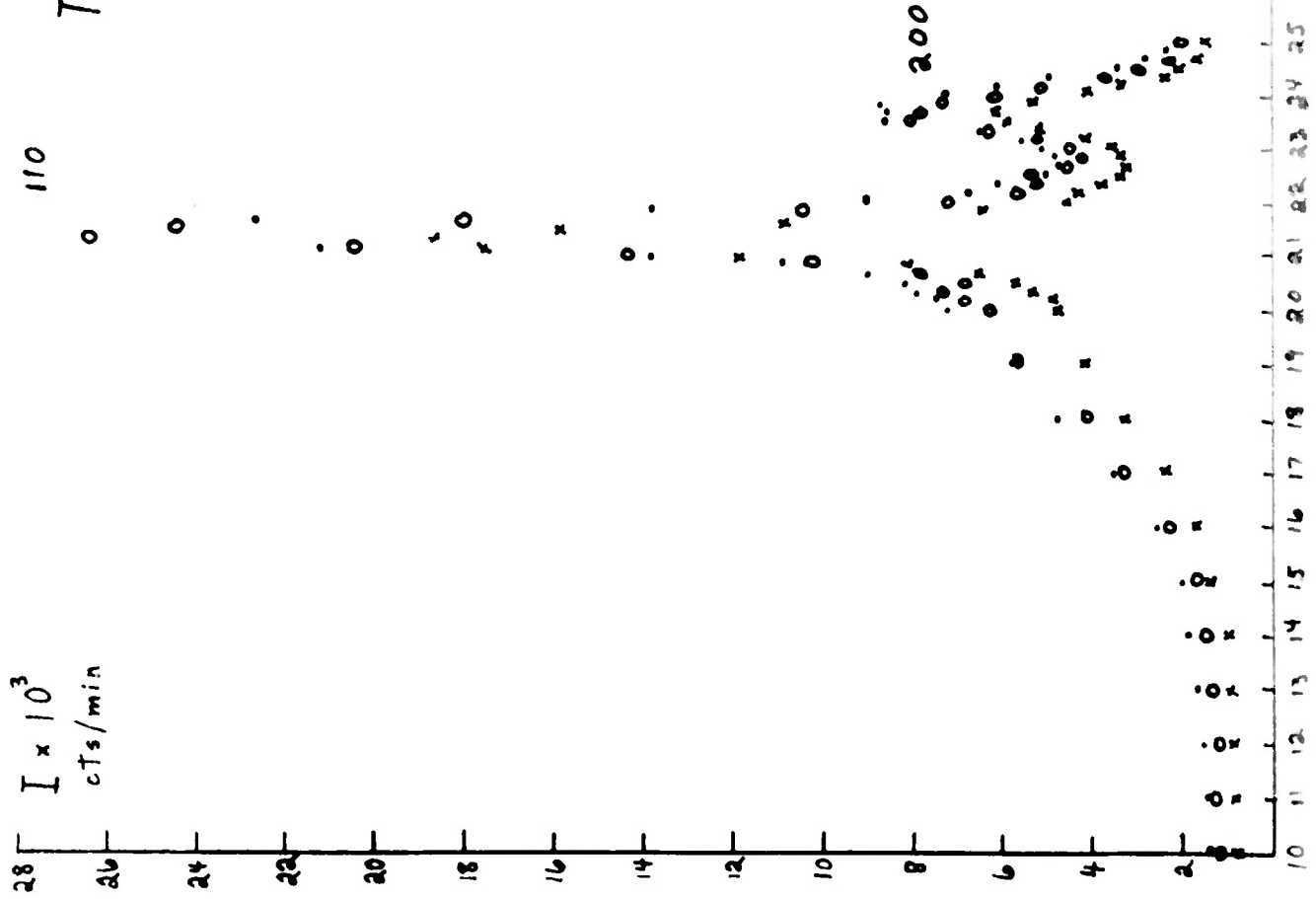
Tilt Angle Scans
Three Polyethylene Samples, 110 Plane

Figure 19

Three Radial Scans - Polyethylene

Sample	Symbol	Thickness
1	.	.0948"
2	o	.0626"
3	x	.0363"

Reflections Indexed by Bunn[5]
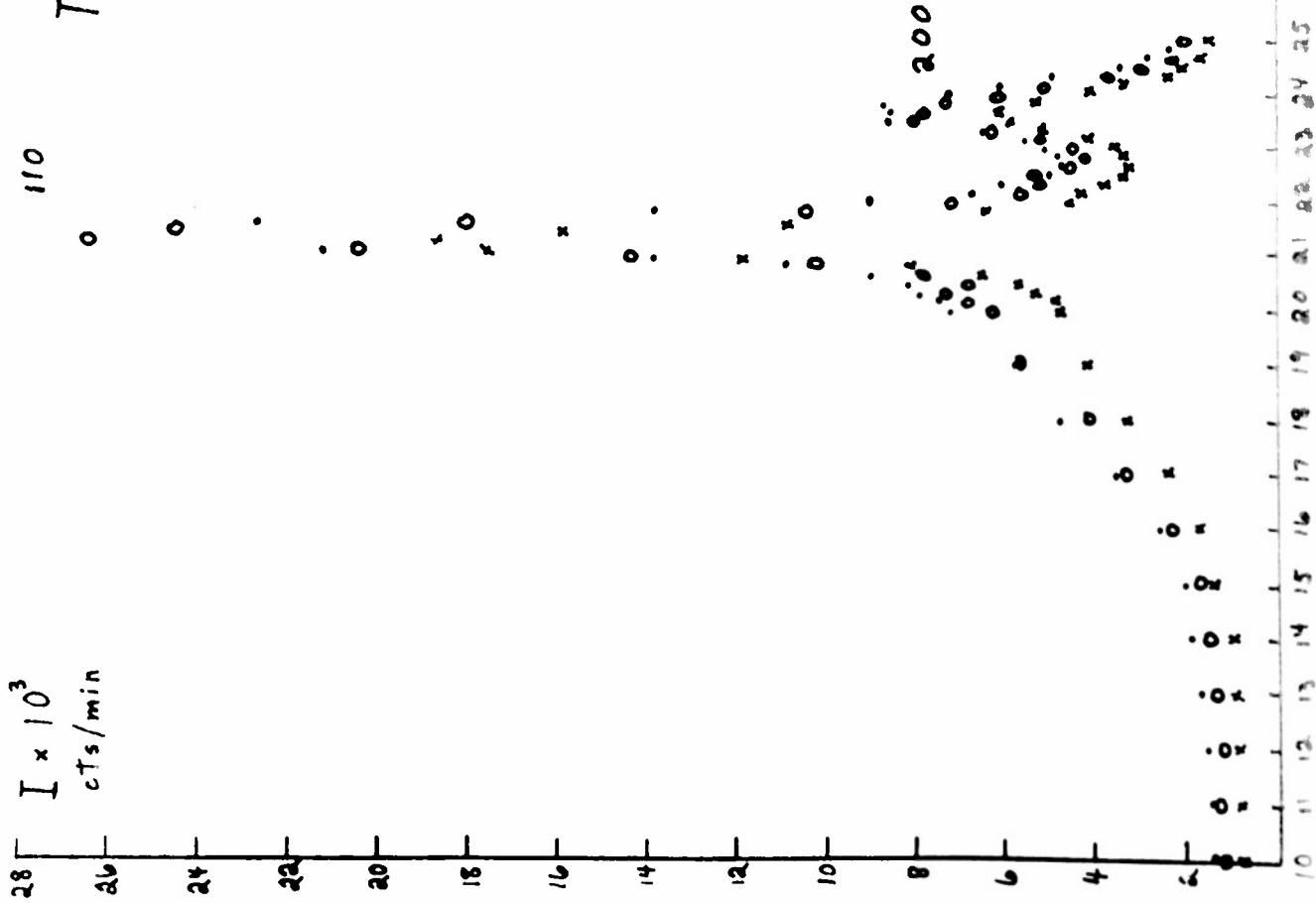
Figure 18
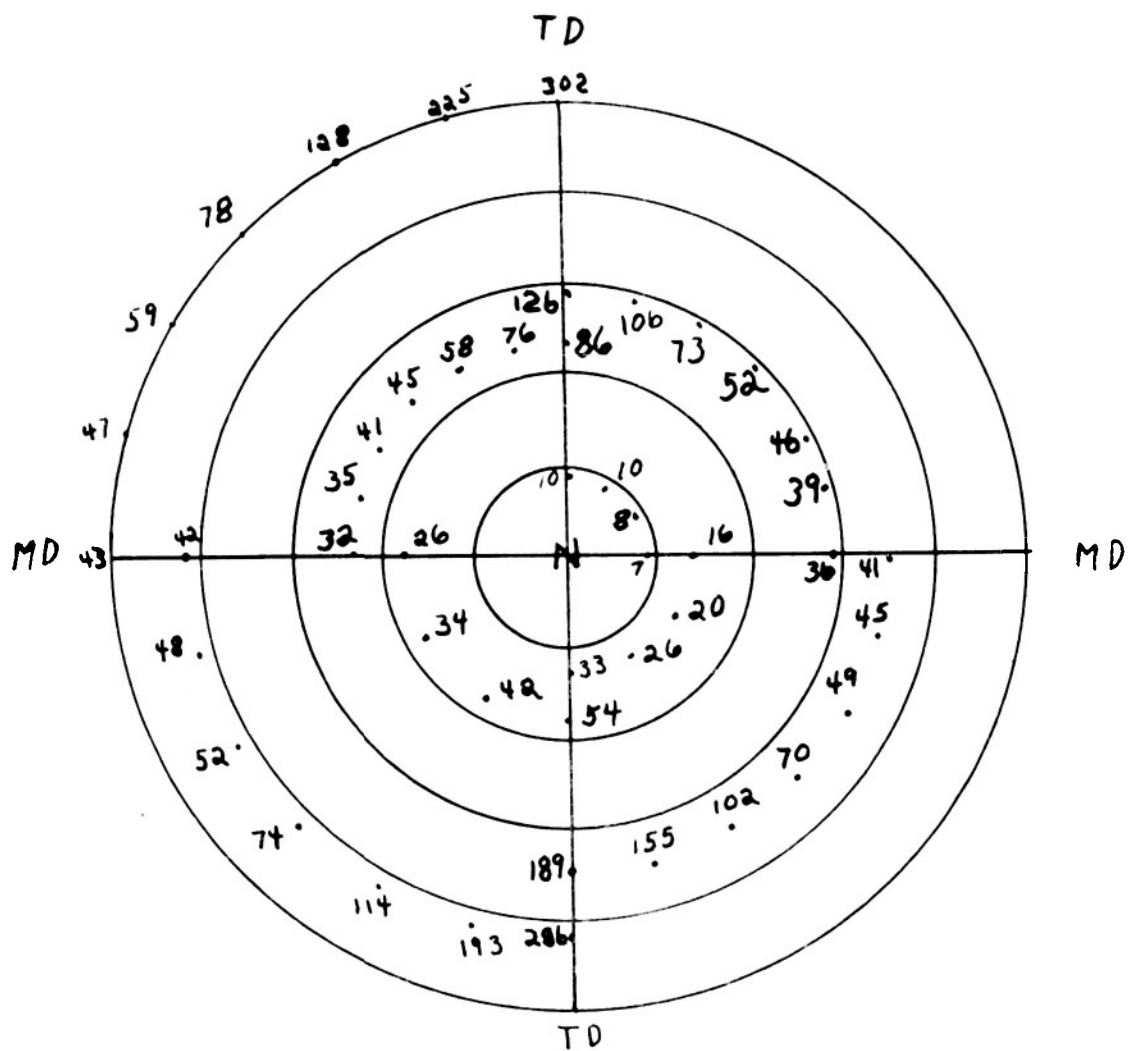
a axis distribution - sample 32G

Figure 20

TD

8000   8000
10,000   10,000
10,000   10,000
8000   8000
5000   5000
4000   4000

MD   N   MD

TD
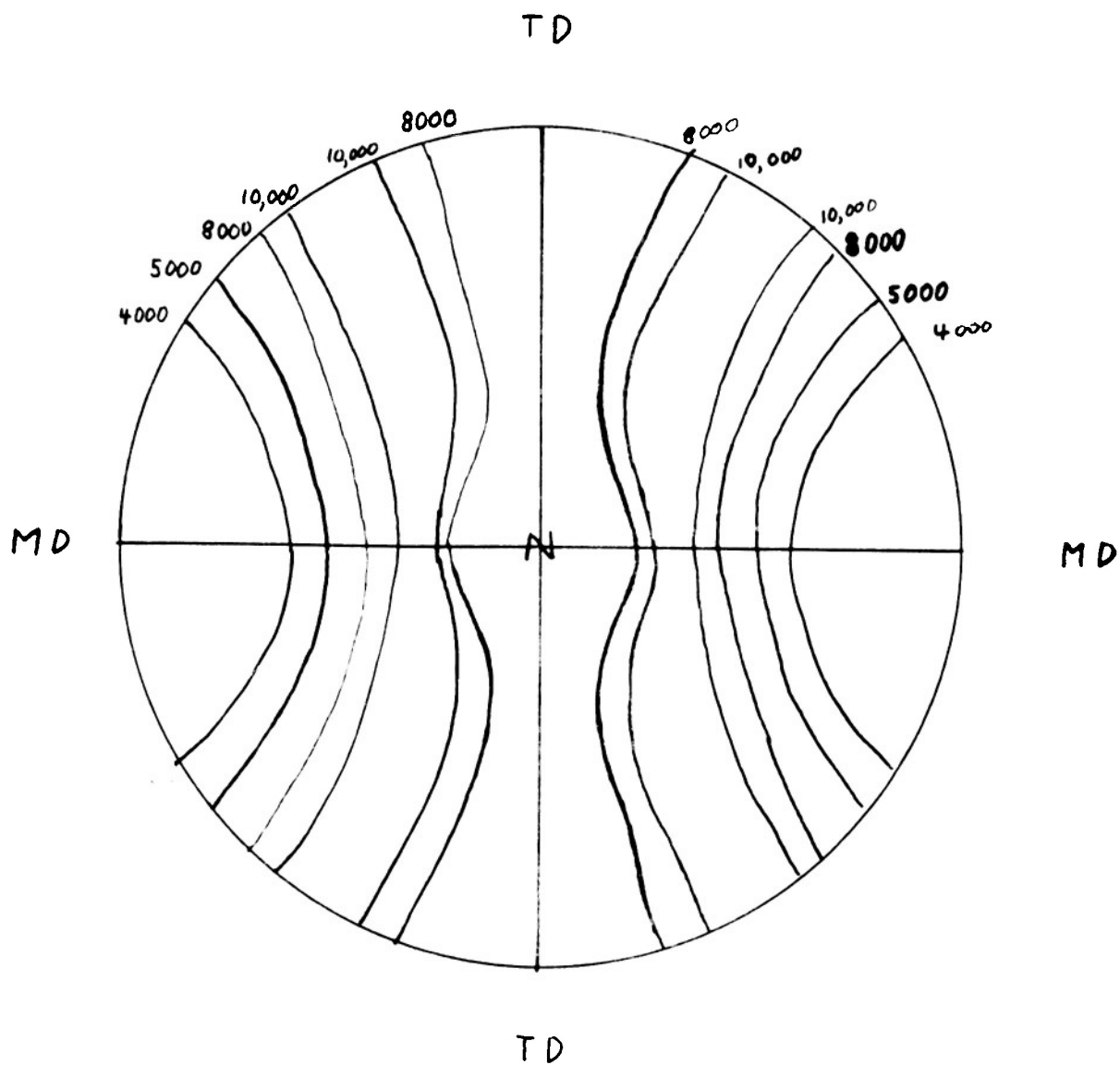
Contour Map
110 intensity - sample 32G

Figure 21

DOCUMENT CONTROL DATA - R&D

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1 ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Polymer Research Institute, Chemistry Department University of Massachusetts, Amherst, Massachusetts 01003	2b GROUP

3 REPORT TITLE

"An Automatic X-Ray Diffractometer for Pole-Figure Analysis of Polymer Orientation"

4 DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Technical Report

5 AUTHOR(S) *(Last name, first name, initial)*

Cooper, C. Richard, submitted by Richard S. Stein

6 REPORT DATE	7a. TOTAL NO OF PAGES	7b. NO. OF REFS
January 15, 1965	86	5

8a CONTRACT OR GRANT NO. NONR 3357(01)	9a. ORIGINATOR'S REPORT NUMBER(S)
b PROJECT NO NR 056-378	78
c	9b OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*
d	AT(30-1)-3003 -2

10 AVAILABILITY/LIMITATION NOTICES

Qualified requesters may obtain copies of this report from Polymer Research Institute, University of Massachusetts, Amherst, Massachusetts 01003

11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY
This report was also supported by the Atomic Energy Commission	Department of the Navy, Office of Naval Research, Boston Branch Office, 495 Summer Street, Boston, Massachusetts 02210

13 ABSTRACT

An automated x-ray diffractometer has been designed and built to obtain pole figure data on polymeric samples. The pole figure goniometer is automatically driven, in a prefixed, constant-increment program, by two pulse motors, allowing the operator to determine the complete distribution pattern for a given h k l plane automatically. The results are punched out on IBM cards; two programs have been written to process this data on the IBM 1620 computer. The first program computes the x-ray intensity at each sample orientation, corrected for absorption, polarization, background, and incoherent scattering. The second program uses this intensity data to obtain locations of constant-intensity contour lines.

DD FORM 1473
1 JAN 64

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
x-ray diffraction						
pole figure analysis						
polymer orientation						
automatic x-ray						

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